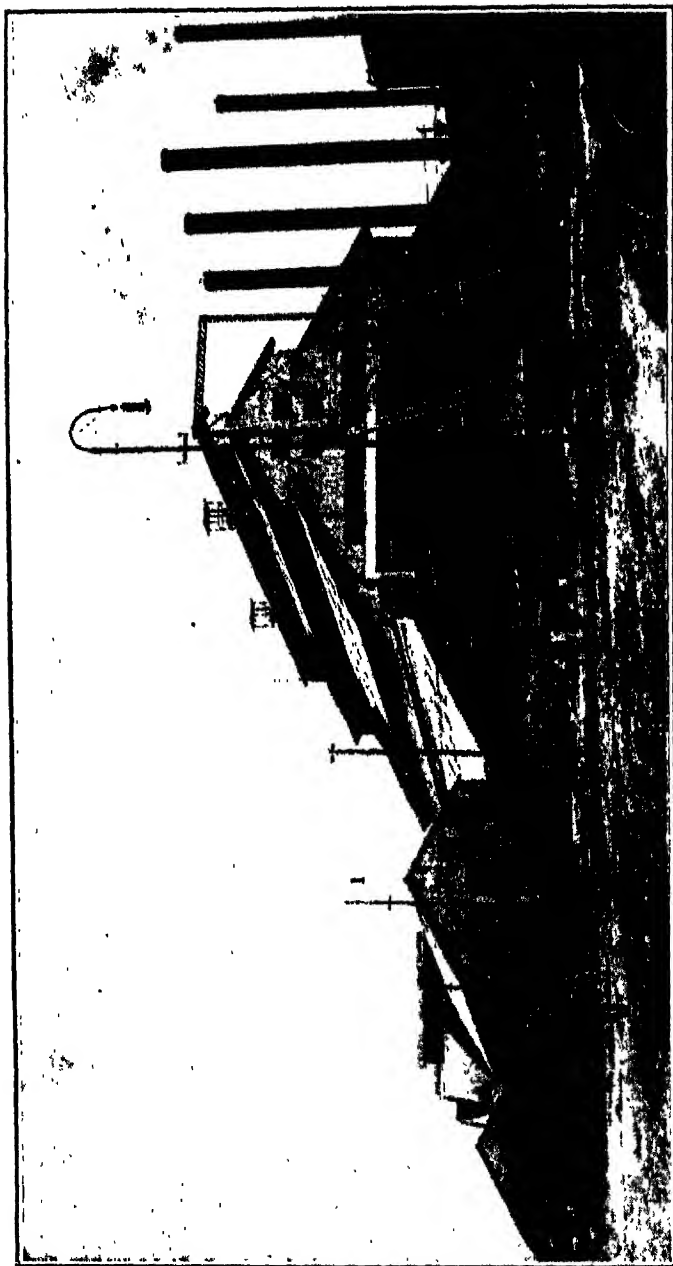


A HANDBOOK  
FOR  
CANE-SUGAR MANUFACTURERS  
AND THEIR CHEMISTS



CENTRAL DELICIAS, CUBA.

*Frontispiece*



FOR  
CANE-SUGAR MANUFACTURERS  
AND THEIR CHEMISTS

BY

The late GUILFORD L. SPENCER, D.Sc.

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SEVENTH EDITION, REVISED, REWRITTEN AND ENLARGED

BY

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DEDICATED  
IN HONOR AND GRATITUDE TO  
M. CH. GALLOIS  
AND  
M. FRANÇOIS DUPONT  
PAST PRESIDENTS  
OF THE  
ASSOCIATION DES CHIMISTES DE FRANCE  
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## GUILFORD LAWSON SPENCER, 1859-1925

GUILFORD L. SPENCER's entire career was devoted to sugar work. His interest in the technology and chemistry of sugar dates from the time when he was a student at Purdue University under Dr. Harvey W. Wiley, with whom he was to be associated later in the Bureau of Chemistry at Washington. After obtaining his bachelor's degree at Purdue in 1879 Spencer went to the University of Michigan for graduate work, receiving an M. S. in 1882. His doctor's degree was granted several years later (1893) by Purdue. From the University of Michigan he went to France to study sugar manufacture under the late Henri Pellet and spent two years in a beet factory where he mastered the practical phases of every station, becoming a trained sugar-boiler and finally passing on to the laboratory. During these two years he came in contact with many of the noted sugar chemists of that day, including Pellet, Sachs, Dupont and others.

The next forty years may be divided into two periods; the first, from 1885 to 1905, when Dr. Spencer was with the Department of Agriculture as Dr. Wiley's assistant and as Chief of the Sugar Laboratory in the Bureau of Chemistry; the second, the last twenty years of his life when he was in commercial work in Cuba as General Superintendent of Manufacture of the Cuban-American Sugar Company. However, throughout the time of his association with the Government he was in constant touch with commercial sugar work and the practical problems of manufacture.

Dr. Wiley was appointed Chief of the Bureau of Chemistry in 1883 and immediately started an extensive study of sugar manufacture (beet, cane, sorghum and maple) in the United States. He appointed Spencer as his assistant, some of whose earliest work with the Bureau was in connection with the investigations instituted by Dr. Wiley into the commercial manufacture of sugar from sorghum by diffusion and carbonation. The work was carried out at Fort Scott, Kansas, under Dr. Spencer's supervision and it was during this work that he had the distinction of boiling the first strike of sorghum sugar ever discharged from a vacuum pan. The succeeding years found this study of diffusion extended to sugar cane at the Warmoth plantation, "Magnolia," in Louisiana, where Spencer demonstrated that from 25 to 40 per cent more sugar could be extracted than by the crude milling methods then in vogue. While at Magnolia he introduced the first filter-press into Louisiana and this may mark the first use of this important piece of machinery in cane-sugar manufacture in the Western hemisphere. The work on diffusion is now of historic interest only, but it is generally conceded that it was through the threat of the new process that manufacturers were forced to develop more powerful mills, which have finally reached an extractive effi-

ciency almost equaling that of diffusion, without the added fuel expense which made the diffusion process impracticable.

During the intervals between sugar crops Dr. Spencer worked in the Bureau at Washington on problems related to sugar chemistry and analysis, thus alternating scientific research with the study of the technology of the sugar house. In 1888 he married Emma Fiske, daughter of Samuel Fiske, inventor of the green bagasse burner and the cane shredder.

In considering the latter half of Spencer's life work, i.e., his twenty years in Cuba, it may be well to quote Dr. Charles A. Browne, who succeeded him as Chief of the Sugar Laboratory and who later succeeded Dr. Wiley as Chief of the Bureau of Chemistry.

"Dr. Spencer's long apprenticeship under Dr. Wiley had convinced him of the wasteful methods of the old hot room, and he began his work in Cuba with the definite plan of producing grain sugar and final molasses in one operation. Success attended his efforts at the very beginning. In March, 1910, he wrote 'the methods are extending rapidly in the Island and I venture to predict that within a few years the string sugars will cease to be produced.' This prophecy was very quickly realized. . . . This splendid result has meant a gain of many millions of dollars to the sugar industry of Cuba, which has profited to an almost equal extent through Dr. Spencer's efforts to produce a dryer, cleaner sugar that would not deteriorate. These two accomplishments—the elimination of molasses sugars and the reduction of losses from deterioration—are lasting monuments to Spencer's work as a raw-sugar technologist."

Of similar importance was his standardization of the analytical procedure, technical control and technical accountancy of cane-sugar manufacture. Practically all well-controlled sugar factories of the Western Hemisphere employ methods along the lines laid down by him.

Dr. Spencer gave to the sugar industry the unusual combination of author, trained scientist, highly specialized technologist and practical sugar-maker. Added to this was a proclivity for designing special laboratory apparatus, and the extent to which he exercised this may be judged from descriptions of his devices which are given in this book. An insight into the activity of his mind and the scope of his work may be gained by the accomplishments of the last five years of his life. During this short period he patented a practical process for the desugaring of cane molasses; designed an electrically operated automatic centrifugal and devised a laboratory apparatus for the calibration of glass flasks that is of such accuracy that it was adopted for use by the Bureau of Standards.

G. P. M.

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# THE MANUFACTURE OF CANE-SUGAR

## CHAPTER I

### ECONOMIC PHASES OF THE SUGAR INDUSTRY

1. **Historical.**—Sugar-cane is generally believed to have originated in Northern India, the earliest mention of it being in some of the legends concerning Buddha about the 4th Century B.C. There was no field culture and its use for several centuries was restricted to chewing the cane and drinking the juice. From India the planting of cane probably spread first into China. Neither sugar nor sugar-cane are referred to in the Bible, the Talmud, or the Koran although all three mention honey several times. The earliest positive evidence of sugar in solid form seems to date from about A.D. 500 in Persia, the original Persian name for white sugar being "kandi-sefid" from which comes our word "candy." "Shekar," or "Shakar," the East Indian word for sugar, shows the origin of our term. In its earliest days sugar was a rare delicacy and was also highly valued for medicinal purposes.

Commercial manufacture and refining developed in Egypt during the 9th and 10th Centuries and the exportation of sugar was an important part of that country's commerce. The culture of the sugar cane had been spread by the Arabs through Northern Africa and Southern Europe at the same time that it was being carried into Java and the Philippines by the Chinese. The Crusaders brought sugar back to France in the 11th and 12th Centuries, after which time the commercial development and use became wide-spread in Europe.

Columbus introduced sugar-cane into Santo Domingo on his second voyage in 1494 and from there it was carried to Cuba and other West Indian, Central and South American sections. The industry did not attain immediate importance here, but by 1600 the production of raw sugar from cane grown in tropical America was the greatest industry in the world at that time. The cultivation in Louisiana dates from 1750.

Sugar refineries were built in Germany, France and England in the 16th Century. The first commercial beet sugar was made about the middle of the 18th Century, the culture and manufacture being largely developed through French initiative toward the end of the century, this branch of the sugar industry being extended to the United States about 1835.

2. **World Production.**—The total sugar produced in the world (both cane and beet) has nearly trebled in the last thirty years (see Fig. 1). The production for the last three years is shown in the following table (Willett & Gray):

CANE SUGAR	Tons, 2,240 Lbs.		
	1927-28	1926-27	1925-26
Cuba.....	4,011,717	4,508,521	4,884,658
United States (La.).....	63,207	42,112	124,447
Porto Rico.....	665,000	562,679	541,485
Hawaii.....	773,000	724,403	705,350
Santo Domingo.....	340,058	303,524	354,720
British West Indies.....	237,000	222,485	215,501
Mexico.....	175,000	181,858	190,282
Argentina.....	421,601	475,695	395,733
Brazil.....	650,000	850,565	676,524
Peru.....	350,000	375,963	275,561
Other countries.....	368,162	361,497	347,922
Total in America.....	8,054,745	8,609,302	8,712,183
British India.....	3,221,000	3,225,000	2,977,000
Java.....	2,359,050	1,959,948	2,278,900
Formosa and Japan.....	691,230	523,054	616,584
Philippines.....	596,033	584,238	436,705
Total in Asia.....	6,867,313	6,322,240	6,309,189
Egypt, Mauritius, Reunion, Natal and Mozambique.....	646,775	619,444	668,514
Australia.....	493,049	415,611	522,344
Fiji Islands.....	95,114	69,071	100,810
Spain.....	9,000	6,719	8,704
Total Cane Sugar.....	16,165,996	16,042,387	16,321,744
BEET SUGAR			
United States.....	965,241	801,246	804,439
Canada.....	27,212	31,422	32,475
Germany.....	1,665,000	1,657,088	1,595,545
Czecho-Slovakia.....	1,240,000	1,043,259	1,497,004
Russia and Ukraine.....	1,471,320	871,020	1,041,903
France.....	870,000	729,082	757,987
Poland.....	565,182	552,553	575,673
Great Britain and Ireland.....	208,114	165,465	51,784
Other Countries in Europe.....	1,939,812	1,853,425	1,933,397
Total Beet Sugar.....	8,951,881	7,704,560	8,290,207
GRAND TOTAL: Cane and Beet Sugar.....	25,117,877	23,746,947	24,611,951

3. Price Range and Price Calculation.—The price of Cuban raws, c & f basis (i.e., cost and freight paid to New York but duty unpaid), for the past thirty years is shown in the chart, Fig. 1. The "duty paid" price or actual

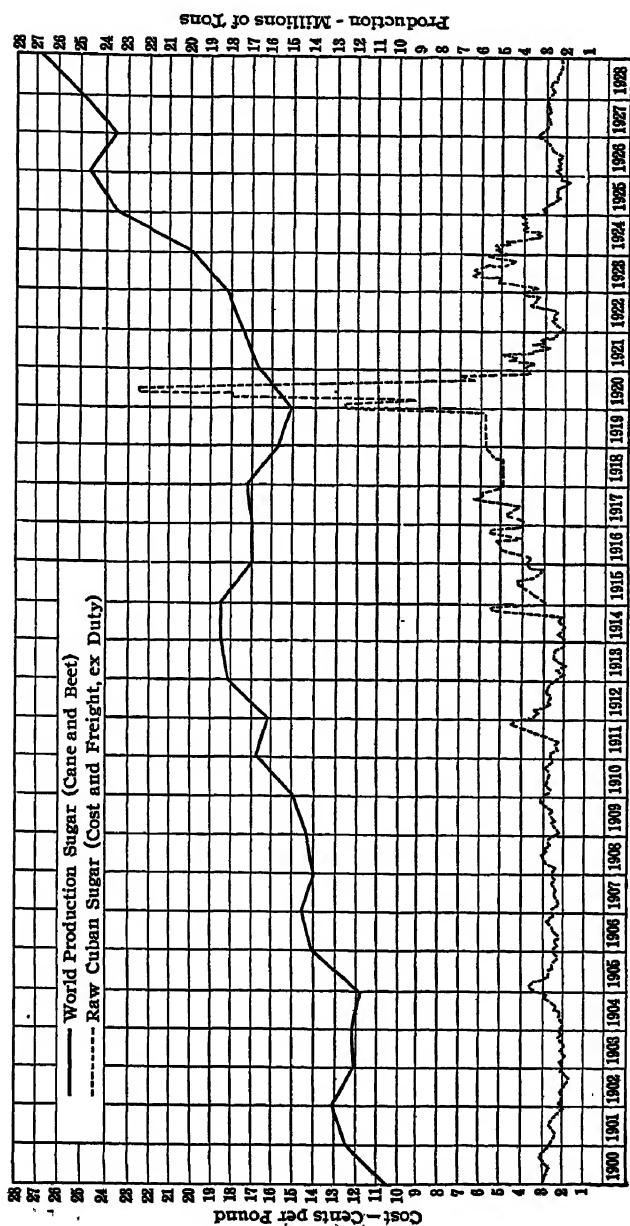


Fig. 1.—Sugar Production and Price Range.

# 4 ECONOMIC PHASES OF THE SUGAR INDUSTRY

cost price paid by the refiners for raws may be obtained by adding the amount of the duty from Cuba as given in the following tabulation:

Basis 96° Test	From Cuba, Cents per Lb.	Other Foreign Countries, Cents per Lb.
July, 1897, to Dec. 27, 1903.....	1.685	1.685
Dec. 27, 1903, to Mar. 1, 1914.....	1.348	1.685
Mar. 1, 1914, to May 27, 1921.....	1.0048	1.256
May 27, 1921, to Sept. 22, 1922.....	1.600	2.000
Sept. 22, 1922, to Date (Feb., 1929).....	1.7648	2.206

The calculation of the duty into the United States (as in force February, 1929) is made as follows: Subtract 75 from the polarization, multiply by 4.6, add 1.24 for full duty sugars; deducting 20 per cent for the preferential duty from Cuba. Thus for 97.2° sugar subtract  $75 = 22.2 \times 4.6 = 1.0212 + 1.24 = 2.2612$  cents, which would be the full duty on 97.2° sugar. Deducting 20 per cent or  $.45224 = 1.80896$  cents, the duty on Cuban raws at 97.2°. Subtracting the duty for 96° test as given above  $1.7648 = .04416$  cent, the "duty increment" used in the price calculation given below.

The Sugar Institute, Inc., adopted the following allowances in March, 1928, for the calculation of the price of raws purchased on a basis of 96° Test Cubas c & f New York:

- 96° to 97°—add 1.50 per cent of the basis price
- 97° to 98°—add an additional 1.25 per cent of basis price
- 96° to 95°—deduct 1.60 per cent of the basis price
- 95° to 94°—deduct an additional 2.00 per cent of basis price
- 94° to 93°—deduct an additional 2.50 per cent of basis price

Fractions of a degree in proportion.

No additional allowance above 98°. No sugar to be delivered below 93° unless on terms mutually satisfactory to Consignee and Seller.

## PRICE ADJUSTMENT ON 97.2° POLARIZATION CUBAN SUGAR PURCHASED ON BASIS 2½ CENTS C & F FOR 96°

	Cents
Basis 96° c & f.....	2.50000
Add for 96 to 97, 1½ per cent.....	.03750
Add for 97 to 97.2, ⅙ of 1½ per cent.....	.00625
Adjusted price, c & f.....	2.54375
Add duty at 96°.....	1.76480
Add duty increment, 96-97.2.....	0.04416
Add insurance.....	0.00520
Cost to refiner.....	4.35791



PRICE ADJUSTMENT ON 97.2° POLARIZATION PORTO RICAN SUGAR PURCHASED  
ON BASIS 4.27 CENTS C.I.F. (COST, INSURANCE, FREIGHT) FOR 96°

	Cents
Basis 96° c.i.f.....	4.27000
Deduct.....	1.77000
Equivalent cost and freight, Cubas.....	2.50000
Add for 96 to 97, 1½ per cent.....	.03750
Add for 97 to 97.2, ⅓ of 1½ per cent.....	.00625
Adjusted equivalent, c. & f.....	2.54375
Add duty at 96° and insurance.....	1.77000
Add duty increment, 96-97.2.....	0.04416
Cost to refiner.....	4.35791

Raws entering England are purchased on 96° Test (cost, insurance, freight) basis, with the following allowances for variations in test:

For each degree above 96°	1½d. (pence) per cwt. added
For each degree below 96°-94°	3d. (pence) allowance per cwt.
For each degree below 94°-93°	4½d. (pence) allowance per cwt.
For each degree below 93°	9d. (pence) allowance per cwt.

The sugar duties for raws entering England (1928) are as follows:

Polarization	Non-preferential Duty		Preferential Duty for British Colonies	
	Shillings	Pence	Shillings	Pence
92-93	7	5.6	4	0.5
93-94	7	8.2	4	1.9
94-95	7	10.9	4	3.3
95-96	8	1.6	4	4.8
96-97	8	4.3	4	6.3
97-98	8	7.0	4	7.7
Exceeding 98	11	8.0	4	9.2
99-100	11	8.0	5	10.0

4. Sugar as a Food.—Refined sugar is practically a chemically pure product consisting of about 99.9 per cent or more of pure sucrose, the remaining tenth of a per cent or less being mostly moisture. Statistics of the United States Department of Agriculture show that at the average market price of 1927 refined sugar gives more food value (in calories) per dollar's worth than any other common foodstuff on the market. The relationship follows: The calories in \$1.00 worth of granulated sugar would have cost \$1.80 in bread; \$2.40 in potatoes; \$3.44 in pork; \$4.07 in milk; \$5.50 in beef; and \$11.10 in eggs. It

is to be understood that granulated sugar is pure carbohydrate while the other products mentioned are more or less whole foods, so the comparison is not one of dietetic value but of the foods as energy producers. The per capita consumption of sugar for various countries during 1927 is listed below:

Australia.....	118.61	Brazil.....	47.18
United States.....	112.21	France.....	43.65
Denmark.....	107.14	Algiers, Morocco and Tunis..	39.02
Cuba.....	100.31	British India.....	27.78
Canada.....	91.93	Hungary.....	27.34
Great Britain.....	90.39	Poland.....	26.68
Ireland.....	76.50	Persia.....	25.75
Switzerland.....	74.74	Spain.....	25.13
Sweden.....	72.09	Japan.....	24.91
Argentina.....	70.33	Mexico.....	24.25
Holland.....	69.67	Egypt.....	23.37
Norway.....	64.59	Italy.....	19.62
Czecho-Slovakia.....	58.86	Russia.....	16.31
Austria.....	57.98	Rumania.....	14.77
Belgium.....	53.13	Bulgaria.....	12.13
Germany.....	52.91	Turkey.....	9.92
British South Africa.....	48.06	China.....	4.19

## CHAPTER II

### RAW MATERIAL

5. **Sugar-Cane.**—Sugar-cane is a large grass belonging to the genus *Saccharum* and ascribed by most authorities to one species, *Saccharum Officinarum*. The endless varieties which occur throughout the tropical and semi-tropical regions of the world are the results of soil conditions, climate, and modes of cultivation. Stubbs in his work "The Sugar Cane" recognized three classes, the divisions being (1) white, yellow and green canes, (2) striped or ribbon canes, and (3) solid colors not included in the first class. Dark-colored canes are usually grown in the sub-tropics while the lighter-colored canes of Class 1 have been favored in the tropics. The usual cane in Cuba is the "cristalina" which occurs abundantly in many sugar countries under various names.

6. **Seedling Canes.**—Up to about forty years ago the flowers were believed to be sterile but the discovery (about 1888) that the cane produces viable seed started a search for new varieties in experiment stations in various parts of the world which has resulted in the production of large numbers of seedlings. The seedlings are crossed with other seedlings and with existing varieties in order to develop certain characteristics. These experiments have resulted in several varieties which are now in broad culture. The new varieties are selected for some particular qualities such as richness in sucrose, resistance to disease, persistence of type, time of ripening, milling qualities, fuel value, color, etc. The first extensive use of seedling varieties was in Java where few of the old varieties are now cultivated.

The seedlings are designated by initials and numbers, the initials indicating the origin, the best known being the famous P. O. J. series (Proefstation Oost Java or East Java Experiment Station). Seedlings originating in Demarara (British Guiana) form the "D" series, while the initials P. R. for Porto Rico, B. for Bardados, H. for Hawaii, etc., are easily recognized.<sup>1</sup>

7. **Selection of Varieties.**—Because of the restricted land in Hawaii and Java a constant search for varieties giving higher yield of *sugar* per acre has resulted in the gradual replacement of old varieties for new,<sup>2</sup> but on this side of the globe except in the British West Indies and Louisiana dependence until very recently has been largely on the so-called "native" canes; i.e., those that have been in cultivation in the locality for many generations.

<sup>1</sup> For an exhaustive description of cane varieties see Arthur H. Rosenfeld, Jour. Dept. Agr. of P. R., Vol. XI, 1927.

<sup>2</sup> See Maxwell, Economic Aspects of Cane Sugar Production. Chapter XI. London, 1927.

Earle<sup>3</sup> says: "It is only through calamities that threaten the very existence of the sugar industry that progress in the knowledge of cane and its culture usually occurs." The terrific inroads of the Mosaic Disease in the Argentine which threatened the extinction of the industry about 1910 were stopped by the introduction of the P. O. J. varieties by Rosenfeld and others. These Java canes, though not immune to mosaic, are highly resistant to its effects, and the crops in the Argentine are now much greater than at any time in the history of the industry. Similarly in Porto Rico mosaic developed to an alarming extent between 1915 and 1918 necessitating the introduction of new varieties by Earle and Rosenfeld. Uba cane, immune to mosaic, was found effective in checking the disease but its low sucrose, high fibre, and other poor manufacturing qualities have caused its replacement by Java and British West Indies seedlings. In 1918 Porto Rican plantings were 98 per cent of "native" canes (Rayada and Cristalina), but today many of the more progressive companies have changed almost completely to seedling varieties with notable increase in yield.<sup>4</sup>

Louisiana used Demarara seedlings for many years, but mosaic, the cane borer and other maladies reduced the yields per acre to an extremely low figure. The P. O. J. canes are now being tried out with indications that they will give the same successful results that attended their use in the Argentine.

Cuba still continues to produce Cristalina almost entirely but mosaic and other diseases are becoming more and more prevalent and may force recourse to seedlings here as they have done in other countries.

**8. Mode of Growth.**—Sugar-cane is propagated by means of the buds that are located at the nodes. Pieces or cuttings of the cane are planted with a very shallow covering of soil or in certain localities are only partly covered, but in this latter event are irrigated. Each bud produces a plant and from each of these there are several shoots or suckers. These form a clump or stool of canes. The cane under suitable soil and climatic conditions is usually planted but once in several years. New plants, termed "ratoons," spring up from the stubble, after harvesting the crop, and produce a second crop and so on. Fiscal or soil and climatic conditions sometimes limit the crop to "plant-cane" or to plant-cane and one or two ratoons.

The length of time that the cane is allowed to grow before harvesting varies greatly in different countries. In Louisiana climatic conditions require that cutting begins after seven or eight months' growth, so that the cane never fully matures; Cuba and Porto Rico average ten to twelve months, whereas in Hawaii the bulk of the crop is allowed eighteen, twenty or even twenty-four months' growth. The ripening of cane depends on many factors, most important of which are the nature of the variety and amount and distribution of rainfall or irrigation. The plant matures with the approach of cool or dry weather, the highest sugar yields being found in countries with a pronounced dry season, while in irrigated countries the distribution of water is suspended for a few

<sup>3</sup> Facts About Sugar. May 7, 1927.

<sup>4</sup> Chardon, "The Varietal Revolution in Porto Rico," Vol. XI, 1927, Jour. Dept. Agr., P. R.

weeks previous to the cutting season in order to ripen the cane. The sucrose content of the stalks increases and the reducing sugars decrease as the plant approaches maturity.

Some varieties of cane "arrow" or flower freely in the tropics while others seldom or never flower. A very few arrows have been noted in Louisiana and then only in exceptionally mild years. It is generally believed in Cuba that a year in which the cane arrows freely is not usually very productive. Such cane, however, is often very rich in sucrose and of low invert-sugar content. It increases in its sucrose content for several months after flowering, and, as is true with other canes, deteriorates as regards the sugar when the rainy season begins. The yield of cane, however, may be small, since the plant grows little taller and heavier after flowering.

Normal sugar-canes are never hollow or partially so. They contain approximately from 87 to 90 per cent of juice and some water, in composition with certain plant constituents (colloid water), that contains little or no sugar. Canes that are abnormal on account of some climatic or other conditions are sometimes hollow, but the proportion of such cane is usually very small.

9. **Harvesting.**—This usually begins long before the cane is considered to be ripe in order to obtain a longer working season. The stalks are cut off close to the ground in harvesting and should be topped just above the highest colored joint. These conditions vary somewhat with the country and whether the fields are irrigated. Where irrigation is practised, the two or three top joints are usually removed for use in planting new fields. Since these joints are of lower sucrose and higher glucose (invert sugar) content than the rest of the stalk, the raw material entering the factory is improved by their removal. Owing to scarcity of labor in Cuba, the canes are often topped too high, thus giving the laborer a larger wage and the farmer more cane, but reducing the return to the manufacturer through a smaller yield of juice in milling and the necessity of grinding the poorer parts of the stalk. It is customary in harvesting in Java to dig the earth from about the plants and cut off the stalks a foot below the surface of the ground. The top joints of the canes are reserved for "seed" in planting. The tops, as stated, are only available for planting when irrigation of the fields is practised.

The sugar content of the cane deteriorates rapidly after the stalks are cut, therefore these should be conveyed to the mills as soon as possible and be immediately ground. The small factory usually has an advantage with respect to the freshness of its raw material, owing to its proximity to the fields, as compared with the large factory. The latter generally has a considerable quantity of cane cut in advance of grinding, which lies exposed to the sun in the fields, otherwise regularity in the delivery of the raw material cannot be assured. This is unquestionably a source of huge loss each year which is preventable in part at least by more systematic harvesting.

Dr. Spencer noted a fall of several degrees in the coefficient of purity of the juice from canes that had been cut and left lying three or four days in a hot climate, exposed to the sun in cars and on the ground. He also stored cane under cover, during cool weather in Louisiana, with no appreciable deterioration in a period of more than two weeks.

The lower leaves of the cane become quite dry as the harvest season advances. These often take fire through accident or intention. It is usual to burn cane fields that contain irritating weeds or when the cane is small and trashy. This burning is to facilitate harvesting and it is practised especially where labor is expensive. The cane is not injured by burning, but it must be harvested very promptly to avoid loss through deterioration, which is accelerated by the burning. The rate of deterioration is greatly increased should rain fall upon the burned cane. The manufacturer agrees in most Cuban cane contracts to receive burned cane up to and including five days without deduction from the price, but in the event of rainfall he may refuse it at any time. The purification of the juice is not usually so readily accomplished with burned as with sound cane, and the heating-surfaces of the evaporator foul sooner. The fine particles of carbon sometimes persist through the manufacture and finally appear in the sugar. It is preferable to grind a mixture of sound and burned cane rather than burned cane alone, since the mixed juices are more readily purified.

**10. Transporting.**—The method of transport of the cane to the factory varies with local conditions. Small factories usually transport their cane in carts or small cars. Portable railways are largely used in the Hawaiian Islands and in Java, but almost not at all in Cuba. The cane is brought to the factory or railway in Cuba in bullock carts. It is flumed to the factories in the Island of Hawaii and in British Guiana is usually transported in punts. The use of flumes and punts complicates the estimating of the percentage yield of juice by the mills. Inferential methods, based upon the analysis of the cane and juice and the weight of the latter, may then become necessary.

**11. Composition of the Cane.**—Sugar-cane varies greatly in richness in different countries, as well as in different localities in the same country, and in different years in the same locality. The sucrose content in Cuba rarely reaches 17 per cent, averaging between 13 and 14 while Louisiana considers 12 per cent as very rich.

The table on next page, showing the composition of the stalks of Louisiana cane at the time of harvesting, November-December, is inserted through the courtesy of Dr. C. A. Browne, Chief of Research Dept. of the Bureau of Chemistry and Soils and formerly of the Louisiana Sugar Experiment Station. The figures are condensed from many analyses of the purple variety of the cane. The composition of the cane varies with climatic conditions, character of the soil, manner of fertilization and cultivation, the age of the cane and its variety.

The juice of the cane contains nitrogenous substances that are more or less objectionable in the manufacture. Fritz Zerban<sup>5</sup> isolated asparagin, glutamin and tyrosin. A part of the asparagin and a still greater part of the glutamin are broken up in the manufacture with the result that aspartic and glutamic acids accumulate in the molasses along with undecomposed asparagin and glutamin. These amids are largely responsible for the ammonia given off during the evaporation of the juice. Acid amids and aminoacids are positive molasses makers.

<sup>5</sup>English Ind. Congress App. Chem. 3, 103.

## COMPOSITION OF LOUISIANA SUGAR-CANE

	Per Cent		Per Cent
Water.....	74.50		74.50
		{ Silica, $\text{SiO}_2$ .....	0.25
		{ Potash, $\text{K}_2\text{O}$ .....	0.12
		{ Soda, $\text{Na}_2\text{O}$ .....	0.01
		{ Lime, $\text{CaO}$ .....	0.02
Ash.....	0.50	{ Magnesia, $\text{MgO}$ .....	0.01
		{ Iron, $\text{Fe}_2\text{O}_3$ .....	Trace
		{ Phosphoric acid, $\text{P}_2\text{O}_5$ .....	0.07
		{ Sulphuric acid, $\text{SO}_3$ .....	0.02
		{ Chlorine, $\text{Cl}$ .....	Trace
		{ Cellulose.....	5.50
Fiber.....	10.00	{ Pentosans. } Xylan.....	2.00
		{ (Cane-gum) } Araban.....	.50
		{ Lignin bodies, etc.....	2.00
		{ Sucrose.....	12.50
Sugars.....	14.00	{ Dextrose.....	.90
		{ Levulose.....	.60
		{ Albuminoids.....	0.12
		{ Amids (as asparagin).....	0.07
Nitrogenous bodies.....	0.40	{ Amido acids (as aspartic)..	0.20
(Total N = .06 per cent)		{ Nitric acid.....	0.01
		{ Ammonia.....	Trace
		{ Xanthin bodies.....	Trace
Fat and wax.....	0.20		0.20
Pectin (gums).....	0.20		0.20
Free acids.....	0.08	(Malic, succinic, etc.).....	0.08
Combined acids.....	0.12	(Malic, succinic, etc.).....	0.12
Total.....	100.00		100.00

A small amount of cane-gums (pentosans) and of fat and wax find their way into the juice during milling; these together with the pectin acids and nitrogenous bodies make up the organic solids, not sugar of the juice. The amount of these organic impurities depends upon the age and variety of the cane and also upon the pressure of the mill-rollers. Their percentage is much higher in the juice from the second and third mills than in the juice from the first mill.<sup>6</sup> Most of the fat and wax, the greater amount of albuminoids, and part of the gums of the juice are removed during the clarification.

The sucrose content of the cane and also the coefficient of purity of the juice vary greatly in different parts of the stalk. The juice of the nodes is of lower sucrose content and lower purity than that of the internodes. A similar difference exists between the lower and upper halves of the stalk

<sup>6</sup> Cane-sugar, Prinsen-Geerligs, 2d edition, 31. Browne, La. Planter, 1904, 32, 49.

The composition of the ash of the juice also varies from year to year, as may be noted in the following table, columns "A":

# ANALYSIS OF THE ASH OF CUBAN CANE JUICES

(Percentages of the Ash)

Factory and crop year.....	A	A	B	C	D	E
	(1913)	(1912)	(1912)	(1912)	(1912)	(1912)
Silica, $\text{SiO}_2$ .....	5.56	11.90	6.82	7.60	6.10	6.46
Iron and alumina, $\text{Fe}_2\text{O}_3$ , $\text{Al}_2\text{O}_3$ ..	19.00	12.48	4.80	8.20	5.44	3.00
Lime, $\text{CaO}$ .....	7.62	10.25	10.77	13.02	5.77	4.70
Magnesia, $\text{MgO}$ .....	6.55	6.83	7.88	7.54	5.17	5.01
Potash, $\text{K}_2\text{O}$ .....	25.15	27.14	32.64	29.30	43.66	46.28
Soda, $\text{Na}_2\text{O}$ .....	5.35	2.32	3.20	4.00	1.88	1.36
Phosphoric acid, $\text{P}_2\text{O}_5$ .....	5.44	7.15	Trace	3.90	4.79	4.21
Sulphuric acid, $\text{SO}_3$ .....	15.02	9.84	10.75	17.94	12.15	4.08
Chlorine, $\text{Cl}$ .....	5.14	5.10	10.95	8.10	11.00	12.90
Carbonic acid, $\text{CO}_2$ .....	2.68	9.59	7.47	3.02	8.22	10.53

12. Reaction of the Juice in the Cane.—The H-ion concentration (pH) of the juice in mature canes has been found to be a fairly constant figure. Paine and Balch of the Bureau of Chemistry and Soils studied the pH of crusher juices in Porto Rico <sup>13</sup> immediately after extraction and found them to be between 4.97 and 5.13 with an average of 5.06. They concluded that the pH number would be a criterion of the quality of the cane being milled. It is of interest to note that the pH of the juice *drops* as the cane matures and the pH of healthy canes in Louisiana where cane rarely reaches maturity is between 5.4 and 5.5. Cane which has been cut and left in the field until the juice has begun to sour, juice from frozen or burned canes, or other abnormal canes will show much lower pH values than given above.

13. Manufacturing Season.—The season begins at greatly varying dates in various parts of the world. In the almost rainless districts where irrigation is practised, grinding may be prosecuted during nearly or quite the entire year. This is true in Peru and in parts of the Hawaiian Islands. The season of manufacture begins in October and November in Louisiana and lasts through December and often into January. The season begins in December in Cuba and in January in Porto Rico. The usual manufacturing period of the West Indies is from December until June, though grinding may continue into September or even longer in parts of the northeast coast of Cuba, with frequent interruption on account of rains in May and the following months. The Hawaiian season begins in November and continues about six months, though climatic conditions in many parts of the Islands permit a very much longer season. The factories of the Dutch East Indies grind during the dry mon-

<sup>13</sup> Sugar. Vol. 29 (1927), p. 206. Vol. 30 (1928), p. 8.



soon, or from about May into November. This corresponds very nearly with the Argentine season.

The advent of the rainy season determines the close of the manufacturing period in the Tropics. The rains not only interfere with the transportation of the cane, but cause it to renew its growth at the expense of its sucrose content.

**14. Purchase of Cane.**—In Cuba cane is sold to the factories without regard to its richness in sugar or the purity of the juice. The grower is allowed 4 to 7 per cent of the weight of the cane in 96° sugar, the percentage varying with the location of the factory and the competition for cane. Porto Rico works on a similar basis, allowing 6 to 7 per cent, but penalizes the seller if the juice shows less than 13 sucrose or 80 purity.<sup>14</sup> In the Hawaiian Islands and Java most of the cane is grown by the factories that grind it. Among the countries that purchase cane upon the basis of its sucrose content are the Philippines, Mauritius and Australia, the payment varying from a half to two-thirds of the sugar recovered by the factory. The difficulties as well as the advantages of purchasing cane on a basis of its analysis are outlined in Chapter XXXVI, page 388 and a method is given there for arranging a scale of prices based on the available sugar in the cane. This method, which was devised by Dr. Spencer many years ago, has been successfully used in recent years by some Louisiana factory owners, the arrangement being that a base price is paid for cane showing an available sugar percentage between certain limits, while a premium is paid for higher percentages and penalties are imposed for lower ones.

<sup>14</sup> Jorge Bird Arias. The Planter Reference Book, July, 1925.

## CHAPTER III

### EXTRACTION OF THE JUICE

**15. Unloading the Cane.**—The cane should be delivered to the mills as promptly as possible after cutting. The loss of sugar every year due to the exposure of the cut cane to the sun in the fields and in cars is unquestionably very large, although difficult to estimate with any degree of accuracy.

The cane is unloaded from the cars and carts by mechanical devices in the well-equipped modern factory. Many forms of such devices are in use, especially in Cuban factories. It is probable that mechanical devices were first used for handling cane in the sorghum-sugar industry.

Where the climate is cool and the cane deteriorates but little in storage, as in Louisiana, derricks are often used. These are usually arranged to lift the cane from the cars or carts and deposit it upon endless conveyors, termed carriers or elevators according to their form, or in large piles for the night work, or to prevent interruption in the cart or railway service. In the latter case, the same derricks are again used in picking up the cane and in placing it upon the carrier.

Raking devices are used in many Louisiana factories and to some extent in Java for pulling the cane from the cars or a platform onto the carrier.

Hoists and dumping devices are very generally employed in Cuba in unloading cars and carts. The hoists usually have a capacity to lift a half or a third of a car-load of cane at a time and drop it into the hopper from which it is carried to the mills by elevators. Chains or cables are passed under the load, in using a hoist, and are attached to a yoke, provided with a tripping device. The breakage of bundles often occasions loss of time in this method. However, the use of hoists greatly simplifies the arrangement of the railway tracks in factories using three milling plants.

Dumping devices are in great favor in Cuba. They discharge the cane quickly and with a minimum of labor. Further, there is little loss of cane in transit. There are two general types of dumping devices. In the one, the car is run upon a platform, which is then tilted and the load is discharged endwise through a swinging door into the hopper of an elevator. In the second type the platform is tilted sidewise. The stakes or sides of the car are hinged at the top and fastened at the bottom with latches, which are released when dumping the load. Hydraulic-power is usually employed in tilting the platform. The platform is hinged at such points that the weight of the load itself causes it to tilt when the water is released from the hydraulic cylinders. With this arrangement very little water pressure is required to return the platform and car to a level position.

After being unloaded the cane is carried from the hoppers to the mills by heavy endless chain elevators, or slat conveyors, which are fitted at intervals with arms which drag the cane with them.

#### MILLING PROCESSES

**16. Milling Machinery.**—Multiple mills are used exclusively in crushing the cane and in expressing its juice. These usually consist of a crusher or shredder, or a combination of these, and from three to seven sets of 3-roller mills. Electric drives for mills are now in quite general use, a separate motor for each 3-roller mill and crusher being the rule. The electrification of milling machinery has resulted in great fuel economy and in many cases, an excess of

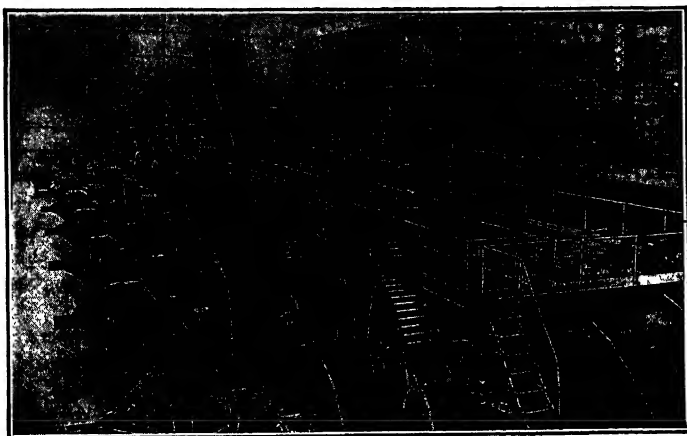


FIG. 3.—Fifteen-Roller Mill and Double Crusher.

bagasse (Sec. 32). A modern mill installation consisting of a Fulton double crusher and fifteen-roll mill, all electrically driven, is shown in Fig. 3. A heavy duty "tandem" of mills such as this, with driving machinery, bed plates, carriers and other accessories makes one of the largest and heaviest machinery combinations in use in any industry. Three such tandems of mills are not uncommon in the larger factories.

**17. Preparation of the Cane for Milling.**—The milling process may be separated into two steps: (1) the preparation of the cane by breaking down the hard structure and rupturing the cells and (2) the actual grinding of the cane. The preparation of the cane is accomplished in several different ways:

- (1) By revolving cane knives which cut the cane into chips; but extract no juice.
- (2) By shredders which tear the cane into shreds; but extract no juice.

(3) By crushers that break and crush the structure of the cane; extracting a large proportion of the juice.

(4) By combinations of any or all of the above means.

**18. Revolving Cane Knives.**—These are the most recent addition to cane-preparing machinery, but as they are always located ahead of any other apparatus in a grinding system they will be described first. Revolving knives are in use in the majority of Hawaiian houses and are rapidly gaining favor in Cuba and Porto Rico.

The earliest cane knives were comparatively slow moving single sets with blades set about 6 inches apart placed at the base of the carrier with a view to leveling the cane mat. To these were added later a second set higher up on the carrier revolving at a speed of 450 to 500 r.p.m. with knives spaced 2 inches

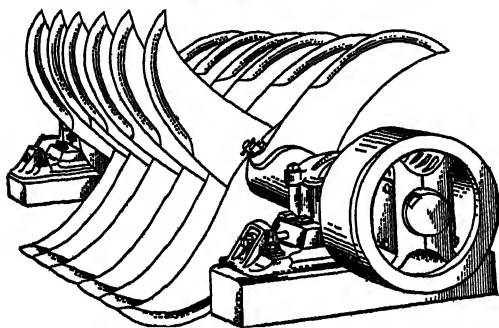


FIG. 4.—Ramsay Cane Knives.

apart. In some countries multiple sets of knives are still used but in Cuba the practice is toward a single set revolving at the speed mentioned above and placed near the middle or toward the head of the carrier. The knives are generally so placed that they reach to within 4 to 6 inches of the carrier slats.

Many different forms of knife blades have been patented, modification being with the idea of facilitating replacement, sharpening and to reduce breakage of blades. In general, the knives are placed spirally on a shaft which is driven by a separate motor, steam-turbine, or high speed engine. The Ramsay swinging knives shown in Fig. 4 are bolted on the shaft in such a way that they are free to swing laterally, so that they may turn aside in case a hard object is encountered in the cane. The hinged arrangement also avoids vibration of the blades and consequent "fatigue" of the metal. A recent reversible type of blade has the front and back edge identical, so that while the front edge is cutting the back edge is being sharpened by the action of the cane sliding past it. The blades are reversed each week, and the self-sharpening action is thus continuous.

The power required to drive the knives is much greater when the blades are dull. For average blade conditions a power consumption of about 1 horsepower per ton of cane per hour is reported.<sup>1</sup> According to Semple<sup>2</sup> the total power to drive knives and mills is no greater per ton of cane than for mills alone without knives.

<sup>1</sup> Proceedings 2d Conference. Int. Soc. Sugar Cane Technologists, Havana, 1927, pages 162-163.

<sup>2</sup> The Planter. Vol. 80 (1928), No. 23.

The revolving knives cut the cane into small chips and therefore cannot be used with ordinary wooden slat cane-carriers. Overlapping steel slat conveyors must be used to avoid the loss of chips, so that the installation of knives in existing mills is made more costly because of this need of carrier replacement.

Knife sets are not used as the sole means of preparing cane for mills but are generally supplementary to crushers or shredders. They are particularly effective in supplying an even feed to the crusher or shredder and mills. They also give increased capacity to the mill train, the reported increases varying from 12 per cent to 20 per cent. More effective use of maceration water and increased sucrose extraction are further advantages that users of knives agree upon.

**19. Shredders.**—The first successful machine for preparing cane for milling was the National cane-shredder, Fig. 5, invented by Samuel Fiske and first

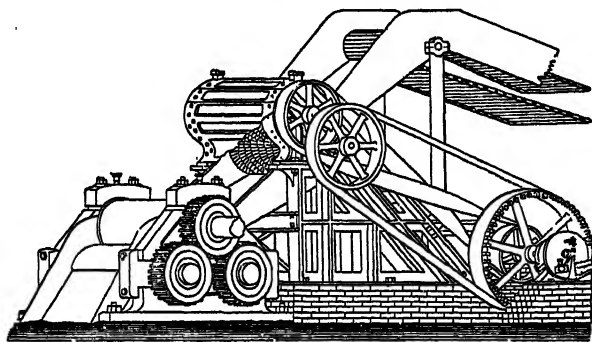


FIG. 5.—National Cane Shredder.

used in Louisiana. This machine consists essentially of two shafts carrying conical cutting disks which dovetail or mesh into one another. These shafts are rapidly revolved in opposite directions and at different rates of speed. The cane is torn into shreds by the disks. The little juice that is separated in the shredding process is immediately reabsorbed.

The National type, although very effective in the disintegration of the cane for milling, has been largely discontinued except in Australia because of operating difficulties and the frequent breakdowns due to " tramp " iron in the cane.

The Searby shredder in rather general use in Hawaii consists of a set of rapidly revolving swing hammers (1200 r.p.m.) which pass between anvil bars on which the cane is beaten and disintegrated into a fluffy mass. The Searby is generally used in conjunction with a crusher, the cane going from the crusher to the shredder and thence to the first mill. Tramp iron does not affect the Searby's operation. This shredder has never come into use in Cuba as the fluffy condition of the cane after shredding is not considered suitable for the heavy feeds run through Cuban mills. Most of the high sucrose

extractions reported from Hawaii (98 per cent and above) are with mills having cane-knives, crusher and Searby shredder in conjunction with 12-roller mills.

The Maxwell shredder is a single cast-iron roll having short hard steel knife blades or teeth held in dovetails in the roll, which operates in conjunction with a crusher or first mill. The toothed roller revolves at a speed of 300 to 400 r.p.m. and the cane is combed into shreds as it issues from the crusher or mill.

The Morgan shredder, which is still in the experimental stage, is described by Semple<sup>3</sup> as follows:

"The cane is first cut in short lengths of 6 inches. As canes in Porto Rico are bundled in the railway cars, all lying the same way, this is readily done by feeding the canes against a series of circular saws, running at 600 r.p.m., spaced 6 inches apart. The sawn cane falls on a belt conveyor, which travels at a high speed, and delivers the cane in a steady stream to the shredder. The shredder is very like an ordinary centrifugal pump with a solid steel disk some 5 feet in diameter in place of a rotor. Hard steel blades, shaped like vanes of a centrifugal pump impeller, are let into each side of the disk, and pass, with very small clearance, similar blades let into the shredder casing. Around the casing is a volute, terminating in a discharge opening about 10 inches in diameter. The cane from the belt conveyor is delivered into both sides of the shredder by small scrolls attached to the shaft and passing along between the revolving and stationary blades is reduced to a fine powder, which is blown at high velocity through an ordinary 10-inch pipe to the mill carrier. The saws are driven by a 125 hp. motor, and the shredder by two 125 hp. motors, all running at 600 r.p.m. Experimentally this installation dealt with about 60 tons of cane per hour with much improved milling results."

20. Crushers.—Crushers, in general, consist of two or more deeply grooved rollers which crush or break the cane, expressing a large part of the juice (from 40 per cent to 70 per cent, depending on type and rates of grinding). They are the main dependence for the preparation of cane for milling and with few exceptions constitute a part of all modern installations. The revolving knives and shredders already described are usually supplementary to crushers. The majority of Cuban factories depend upon crushers only in their grinding trains but with the increasing emphasis on higher sucrose extraction, together with the large capacities always desired in Cuba, revolving cane knives are coming into more general use as preliminary preparation for the cane mat entering the crusher.

The Krajewski crusher was invented by Krajewski in Cuba after the introduction of the shredder into this island. A crusher roll is shown in Fig. 6. The rolls are driven slowly by gearing, the recent tendency being toward faster rates of speed than for mill rolls. In the Hawaiian Islands Krajewski rolls have been grooved with Messchaert grooving (Sec. 22) to permit the free flow of juice.

A second type of crusher is the Fulton, which differs in its cutting or crushing surface quite radically from that of the Krajewski crusher. The cutting teeth are V-shaped and are arranged spirally ranging from 1.75 inches to 3 inches pitch, with the spirals working from opposite ends of the rolls, as is well

<sup>3</sup> *Loc. cit.*

shown in Fig. 7. There are also grooves separating the teeth into groups, and scrapers are provided to prevent clogging the teeth.

A further improvement is the double crusher shown in Fig. 8 in which the cane passes through two crushers before entering the mills. The pre-crusher has much larger teeth, 3 inches or even 4 inches pitch being used with 42-inch diameter rolls. A triple crusher is in use in one large factory in Cuba. A marked tendency toward increase in peripheral speed of crusher rolls has been

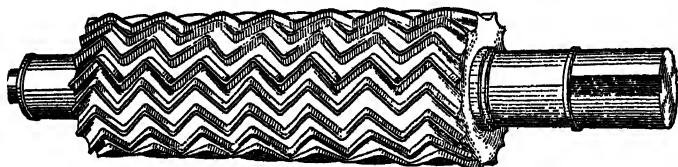


FIG. 6. Krajewski Crusher Roll.

under way so that speeds of 45 or 50 ft. per minute are common as compared with 25 to 30 ft. ten or fifteen years ago.

Three-roller crushers are in use in Hawaii and are favorably reported upon. Such a crusher is essentially a three-roller-mill having deep V-shaped annular grooving ( $1\frac{1}{4}$ -inch to  $1\frac{1}{2}$ -inch pitch). According to McAllep<sup>4</sup> about 75 per cent of the juice can be extracted by one 3-roller crusher preceded by cane knives as against 40 per cent to 50 per cent for single crushers and 65 per cent for double crushers.

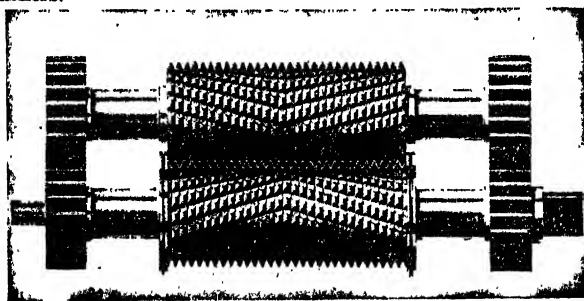


FIG. 7.—Fulton Crusher Rolls.

**21. Mill Trains.**—Present-day mills always have three rolls, as is shown in Fig. 5. The bottom roll, where the cane enters, is termed the "cane roll," and that opposite the "bagasse" or "discharge-roll." The two bottom rolls are usually rigidly fixed in position and the top roll is controlled by an hydraulic ram and is so arranged that it may rise and fall or "float" with variations in the feed of the cane. Hydraulic pressure is applied to the bagasse-roll by certain builders. A ram is shown in cross-section in the top-roll cap in Fig. 10. The crushed cane, now called "bagasse," is passed from one pair of rolls to the

<sup>4</sup>Proc. 2d Conf. Int. Soc. of Sug. Cane Tech. Hayana, 1927.

next by a curved plate also shown in cross-section in Fig. 10 variously termed a turnplate, knife, dumb-turner, trash-turner, etc., according to the country

in which the mill is used. This is supported by a heavy steel turn-plate-bar.

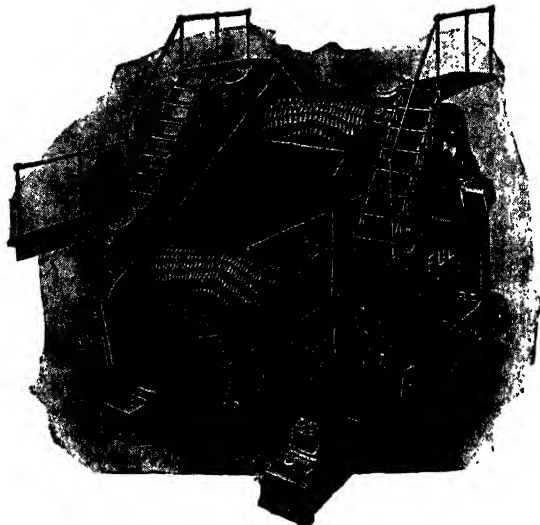


FIG. 8.—Double Crusher.

The mill-rolls are supported in massive castings termed housings or mill-cheeks. The older types of housings are shown in Fig. 5 and more recent models in Figs. 9 and 10. The crown wheels by which the bottom rolls are driven from the top roll are shown in Fig. 5. The driving-engine is connected

through flexible couplings and gearing with the top roll of the mill.

A recent Fulton housing shown in Fig. 9 dispenses with the king-bolts which one may note projecting above the top-roll caps of other types in Figs. 5 and 10. The latter shows a cross-section of a Mirrlees - Watson mill.

#### 22. Mill Grooves.

—Mill-rolls are cast of an iron mixture that will remain rough, or acquire a "grain" with use, to facilitate the feeding of the cane and bagasse. In general, all rolls have peripheral V-shaped grooving,

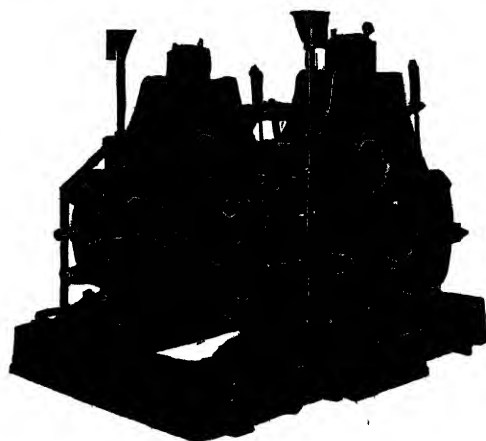


FIG. 9.—Fulton Heavy Duty Mill Housing.

the size of the grooves having been greatly increased to reduce the slipping of the roll upon the bagasse. Where formerly the grooves ranged from four to



six to the inch modern practice now prescribes a minimum of  $\frac{1}{4}$ -inch grooves on the rolls of the last mills. The first and second mills carry grooves of 1 inch

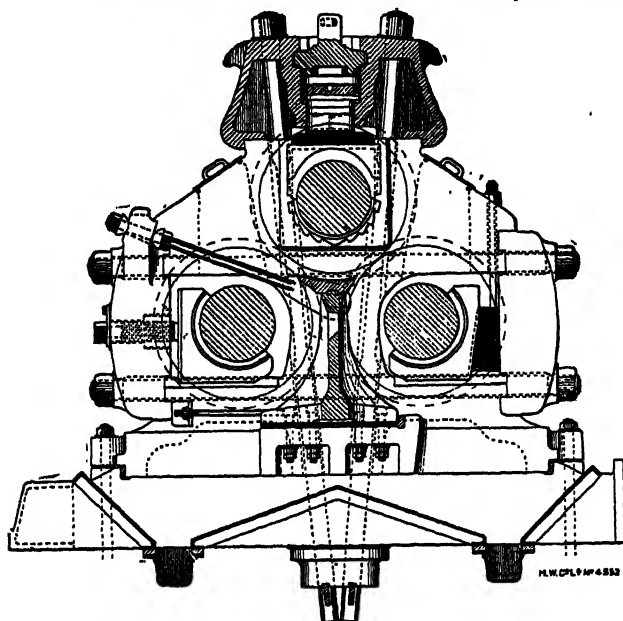


FIG. 10.—Cross-Section of Mirreles-Watson Mill.

and the top roll is grooved longitudinally in the same way as the Fulton crusher roll. The contained angle of the annular grooves is usually  $55^\circ$ . In some very large installations working to high capacities  $1\frac{1}{2}$ -inch and even 2-inch grooves are used on the earlier mills of the train. Such heavily grooved mills are practically the equivalent of the 3-roller crusher used in Hawaii.

The Messchaert juice grooves shown in Fig. 11 are now in general use on feed-rolls throughout the world except in Java. The grooves are usually  $\frac{1}{4}$  inch wide,  $1\frac{1}{4}$  to

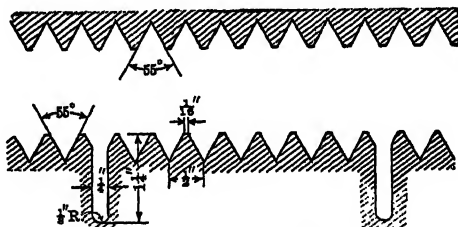


FIG. 11.—Messchaert Mill Grooves.

$1\frac{1}{4}$  inches deep, the pitch varying from 2 inches to 4 inches, with 3 inches the general practice. Special scrapers are used to keep the grooves free of bagasse. These grooves were developed in Hawaii about 1912 and have been the means of improving sucrose extractions and increasing mill capaci-

ties, since they provide a very free exit for the juice and practically eliminate slipping and the consequent mill vibration. An unlimited quantity of saturation water may be used without slipping or reduction of grinding capacity and the mill inlets and outlets may be materially reduced in size, in some instances nearly one-half.

Hawaiian practice, as well as that in some Cuban mills, also provides the discharge or bagasse roll with Messchaert grooves  $\frac{1}{4}$  inch wide, the moisture content of the bagasse being materially reduced by this grooving of the discharge roll.

Another Hawaiian invention is the Hind-Renton grooving. This has been tried out in a milling plant that obtained an average for the crop of over 97 per cent extraction (sucrose extracted per cent sucrose in cane). The pitch is two grooves per inch and the groove angle is  $30^\circ$  instead of the usual angle of about  $55^\circ$ . It is claimed that the juice flows out through the lower part of the groove and that the bagasse wedges itself above it as a boot in a "boot-jack."

Steel shell rolls were used in Australia with this grooving and worked well so far as grinding was concerned, but were unsuccessful because casual iron would bend the teeth which would in turn break off the teeth of the trash knives.<sup>5</sup>

**23. Pressure on Rolls.**—The pressure on the top roll is usually regulated by means of hydraulic rams (see Fig. 10), except in Java.

The hydraulic pressure applied on the top roll varies with the length of the roll, the strength of the mill and the quantity of cane to be ground in a given time. The pressure is also varied with the position of the mill in the series or "tandem."

Practice varies as to the loading, but this approximates 150 to 250 tons hydraulic pressure on the crusher and up to 500 tons or above on other mills. A Cuban mill consisting of cane knives, single crusher and seven 3-roller mills (rolls 7 feet in length) with a record of nearly 250 tons of cane per hour (Central Vertientes) reports pressures as follows; <sup>6</sup> crusher 170–250 tons; 1st mill, 500; 2d mill, 450; 3d mill, 480; 4th and 5th mills, 500; 6th mill, 520, and 7th mill, 550 tons. There is much difference of opinion and practice as to whether higher pressures should be put on the last mills (as in the instance cited) or whether the earlier mills should get the heaviest pressure.

Many engineers advocate the heaviest possible pressure on the first mill in order to rupture and disintegrate the cane as thoroughly as possible to prepare it for the maceration water, while others insist that the last mill pressure should be highest to insure as low a moisture content in the bagasse as is practicable. Still others carry full pressure throughout the whole train.

The hydraulic regulation of the top roll has a two-fold purpose, viz., the protection of the mills from serious damage should a piece of metal fall into them or in the event of a too heavy feed of cane, and the regulation of the opening between the rolls to suit variations in the quantity of cane or bagasse passing through them. The hydraulic pressure now carried is so great that a

<sup>5</sup> R. Clayton. Proc. 2d Conf. Int. Soc. Cane Sugar Tech. Havana, 1927.

<sup>6</sup> Scharnberg. Facts About Sugar. Jan. 14, 1928.

piece of metal may sometimes bury itself in the shell without raising the roll sufficiently to afford protection.

The use of very strong cast-steel housings or mill-cheeks has enabled manufacturers to dispense with king-bolts, Fig. 9, or to use very short bolts that extend only part way through the housing (Honolulu Iron Works). This arrangement permits the use of hydraulic rams of large diameter with consequent increase of life in the packing leathers.

**24. Mill Settings and Speeds.**—The mill "setting" or the adjustment of the openings between the rolls and the relation of the turn-plate to the rolls, varies greatly in different factories and with the rate of grinding and the quality of the canes and the grooving of the rolls. The setting is also somewhat modified when hydraulic-pressure is not used on the top roll, or when it is applied to the bagasse-roll.

It is outside the scope of this chapter to give figures on mill settings since these vary so widely with differing conditions that figures would be valueless without all correlative data. Sharnberg<sup>7</sup> gives full details for the single crusher and seven 3-roller mill combination referred to above showing openings varying from  $2\frac{1}{2}$  inches on the crusher (3-inch pitch) to about  $\frac{3}{8}$  inch on the discharge roll of the last mill, these openings being measured from the tip of one tooth to the base of the corresponding groove. It may be noted that the opening between the turn-plate and the top roll is gradually enlarged from the inlet to the outlet end. This enlargement permits the bagasse to expand after the first pressure and facilitates the passage of this material and the escape of the juice.

The speed at which a system of mills is driven is usually expressed in feet per minute of the periphery of the rolls. Practice varies greatly in different countries in regard to the speed of the rolls. This ranges from as low as 12 feet or less in Java to as high as over 40 feet per minute.

The Cuban practice has been toward higher mill speeds with 30 feet per minute the general rate in good modern mills. As already stated, crusher speeds range above 40 feet per minute, peripheral speed.

**25. Cane and Bagasse Carriers.**—The cane elevator is generally driven by independent engines or motors rather than from the milling machinery. This results in greater uniformity in the delivery of the cane to the crusher and thus gives a more even feed throughout the whole set of mills.

Between the different mills are "intermediate" carriers, the commonest in Cuba being an endless-chain "apron" or wooden-slat conveyor. These conveyors tend to foul through the souring of juice on chains, slats, etc., besides being a source of mill stops because of breakage. This type has been entirely superseded in Hawaii, and to some extent in Cuba, by either the Meinecke chute or Ewart drag conveyor. The Meinecke type (Fig. 12) consists of a covered steel chute rising at an angle of about  $35^\circ$  from the discharge roll of the mill and then dropping sharply to the feed opening of the next mill. The bagasse is forced up the chute by the succeeding bagasse that is discharged from the mill and falls into the next mill by gravity. The maceration

<sup>7</sup> *Loc. cit.*

water was formerly applied at the top of the chute but is now put on just as the bagasse is emerging from the mill.

The Ewart carrier is a steel trough which the bagasse is moved by on endless chain drag travelling above the trough. Both the Ewart and Meinecke methods make for better mill sanitation and are easier to maintain than the older type slat-conveyors.

**26. Extraction of the Juice.**—Having described the mechanical equipment of the milling-plant, we will now consider the crushing of the cane and the extraction of the juice.

Under the usual conditions, the cane parts with more than 60 per cent of its weight of juice in its passage through the crusher and the first mill. In other terms, upwards of 70 per cent, more in some cases, of the sucrose in the cane is extracted. The woody residue of the cane from the first and subsequent mills is termed "bagasse" by the Americans and "megasse" by the English. As

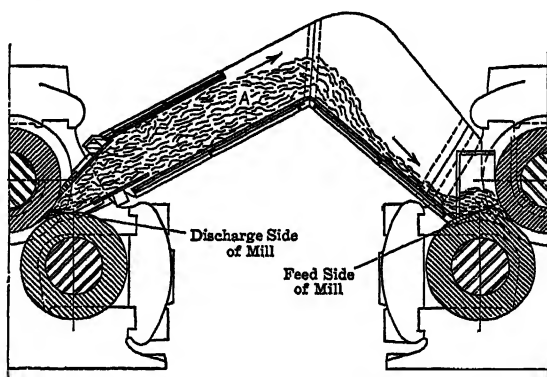


FIG. 12.—Meinecke Intermediate Bagasse Chute.

the bagasse progresses through the mills it is compressed more and more, each time parting with some juice, until finally under good working conditions it leaves the mills containing 50 per cent of woody fiber.

Few factories conduct their mill work as above without adding water to the bagasse after each mill to dilute the remaining juice and thus increase the extraction. This use of water is termed saturation, imbibition or maceration. These words are usually used synonymously.

**27. Maceration.**—The maceration process, properly speaking, is one in which the bagasse is passed through a bath of thin juice or water before regrinding, with a view of diluting the remaining juice. This process is little used except in Australia and the word "maceration" has come to have the same meaning as saturation, etc.

The saturation may be single, double or compound. Single saturation uses water only; double saturation uses water and the thin juice from the last mill or mills, while compound maceration uses water and the juices from two

or more of the last sets of mills, applying these juices separately to the earlier mills of the train.

The water is sprayed upon the bagasse, in single saturation in a 9-roll plant, as it emerges from the second mill. In double saturation, the water is applied as above and the thin juice extracted by the last mill is pumped back upon the bagasse as it emerges from the first mill. Working with four mills, with single saturation, the water is applied after the second and third mills. Compound saturation may be practised when four or more mills are in the series or "tandem." This method is illustrated in the diagram, Fig. 16. The thin juice extracted by the third mill is pumped back upon the bagasse from the first mill and that of the last mill upon the bagasse from the second mill. Water is applied to the bagasse from the third mill. The juice from the crusher, first and second mills enters into the manufacture. The application of the water is modified to meet the needs of other combinations of rolls exceeding 12 in number.

Maceration water is sometimes applied to both the upper and lower side of the blanket of bagasse. The application to the upper surface of the bagasse does not usually penetrate the lower layers. It is preferable in applying the water from above to do so just as the bagasse emerges from between the rolls so that it will absorb the water in expanding. In this way it acts as a sponge that has been compressed.

General practice is to apply the water to the bagasse as it issues from the two mills next from the last one. A method of double saturation in vogue in Cuba is shown in Fig. 13 for a double crusher and 15-roll mill. It will be seen that the water is sprayed on the bagasse coming from the third and fourth mills; that the juice from the fourth and fifth mills is mixed and sent back to the bagasse coming from the first and second mills. Such a system is simpler than compound saturation as it requires only one set of pumps, strainers, etc., for the thin juice, but it is not so efficient as compound saturation in which the juice from the fourth mill would go to the first mill bagasse and the juice from the last mill to the bagasse from the second. Compound maceration is general in Hawaii and Java and many Cuban factories also make use of the system.

There is always danger of fermentation in the rehandling of thin juices and great care must be taken to prevent souring and fouling of juice channels where these methods are used. The mills must be shut down at frequent intervals for a thorough cleaning. Few Cuban factories can spare the time to wash down mills and tanks thoroughly more often than once a week.

The percentage of maceration water used varies widely with the country, the capacity of the mills, the character of the cane and the relative costs of fuel and sugar. The introduction of juice grooves and heavier grooving on Cuban mills has permitted the addition of more water with larger capacities; while

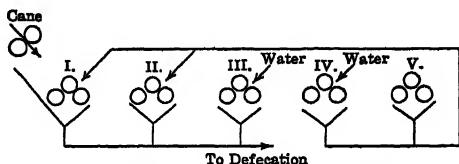


FIG. 13.—Diagram of Double Saturation.

various fuel economies, notably through electrification, have made the use of this increased maceration economical. Twenty-five per cent water is now common practice in the larger mills where only 10. or 15 per cent was used formerly. The average for the island is still about 15 per cent. Hawaii and Australia range up to 35 and 40 per cent, and Java uses 15 to 20 per cent.

Australia uses "true maceration" or "bath" maceration, the system being made practicable by having a space of from 40 to 60 feet between the mills. The bagasse is drawn through a V-shaped closed casing by a drag conveyor at such a rate that the bagasse is in the macerating liquid in the casing (either hot water or hot thin juice) for from ten to twenty minutes. Sucrose extractions of 99 per cent with grinding rates up to 60 tons per hour are reported. This practice is very old and has been discarded in all other countries because of difficulty in keeping the thin juice from souring. Reports from Java are that the system is being tried out again with good results in that country.

Tests by many investigators lead to the conclusion that the mill extraction is practically the same whether the maceration-water be used hot or cold. The matter of the quality of the water and some fuel economy usually dictates the use of warm water. Alkaline water should not be used in white-sugar manufacture. The hot water is derived from the surplus of return water, from evaporator-coils, etc. (so-called "sweet water") over the requirements of the steam-plant, and is therefore very pure distilled water. Those of the heat units that pass with the saturation water into the juice are largely economized. There is also a slightly increased evaporation of moisture from the bagasse in transit to the fires, as compared with that obtained with cold saturation.

The saturation water never completely penetrates the bagasse. Neither the physical condition of the bagasse nor the time element, *i.e.*, the duration of the contact with the water, permits complete penetration. Manifestly the nearer we approach this ideal condition, complete penetration, the better the extraction of the sugar.

Modern mill practice is toward disintegration of the cane as completely and as early as possible with a view to preparing it for the penetration of the maceration liquids. For this reason the use of cane knives, shredders, multiple crushers and combinations of these has increased so rapidly. As stated under the section dealing with hydraulic pressures a large proportion of engineers use the heaviest possible pressure on the first mill of the tandem in order to break up the cane thoroughly for the maceration process.

If all the juice-cells of the plant are ruptured in the shredding or crushing process and the first grinding, it is evident that when the water is applied to the bagasse, if the time element be sufficient, it will penetrate it and dilute all of the juice it contains. The time element, however, in practice is so short, and so many of the cells escape rupture, that only the superficial portions of the juice are diluted.

Dr. Spencer demonstrated by laboratory experiment as follows, that very long contact with water is required for the dilution of all the residual juice in the bagasse: A sample of bagasse from thoroughly crushed cane was heated with water in the proportion of 5 parts of bagasse to 45 parts of water, and the

temperature was maintained near the boiling-point one hour. The water and bagasse were then thoroughly mixed and the dilute juice was strained off, using moderate pressure. The residual juice was expressed with a laboratory cane-mill, using very heavy pressure, and the two samples of juice were separately analyzed. The percentage of sugar in the juice extracted by the mill was very perceptibly larger than that in the juice obtained by straining. This experiment was repeated several times with like results. These experiments show that it is not practicable in milling to dilute all of the juice in the bagasse with the saturation-water, and that a factor depending upon the time element and the efficiency of the mills must be applied in estimates of the water actually utilized.

The following figures are from records of actual milling:

COMPARISON OF MILL-JUICES

Source of the Samples	Juice Analyses		
	Degree Brix	Per Cent Sucrose	Coefficient of Purity
Crusher.....	21.4	19.5	91.1
First mill, front roll.....	21.2	18.8	88.7
First mill, back roll.....	20.7	18.1	87.4
Second mill, front roll.....	18.7	15.8	84.5
Second mill, back roll.....	19.8	16.5	83.3
Third mill, front roll.....	7.5	6.1	81.3
Third mill, back roll.....	9.4	7.5	79.3

Water was sprayed upon the bagasse as it emerged from the second mill. The upper layers of the bagasse were well saturated, but the lower ones received much less water, as the latter was partly absorbed and comparatively little penetrated to the lower layers. It is evident from the analyses that the water did not uniformly dilute the juice in the bagasse.

*Influence of the Structure of the Cane on Milling.*—The structure has a marked influence on the mill results. With very efficient milling certain canes yield bagasse containing 50 per cent woody fiber (marc) and 45 per cent of moisture; others, when ground with the same mills and mill-setting and apparently the same efficiency, give bagasse containing 45 per cent of fiber and 50 per cent of moisture. These conditions have been observed in Java when grinding the varieties Nos. 247 and 100.

In his study of "The Influence of the Structure of the Cane on Mill Work" <sup>8</sup> Noël Deerr says: "It not infrequently happens that while the fiber remains of constant percentage, the extraction varies largely, the milling conditions remaining the same. Such variation can be readily understood on the assump-

<sup>8</sup> Bul. 30, Hawaiian Sugar Planter's Expt. Sta., 41.

forces itself through the screen because of the slight head, this strained juice also being discharged into (K). As the drum revolves the strained material on the outside of the screen falls into the tank and is caught by the paddles (H) and carried to the discharge lip (P), where suitable scrapers (O) engage the paddles and remove the strained material from the system. Arrangements are provided for steaming out the strainer while in operation. This fine straining of juice has the advantage of removing the bagasse fiber before it has undergone the solvent action of heat and lime, but it also has the disadvantage of making the cachaza or mud-scums harder to filter-press because of the lack of this fiber which acts as a filter-aid. The addition of more lime has been suggested but this is known to be detrimental to the character of the press juices. An artificial filter-aid, such as kieselguhr, or the return of some of the "cush-cush" or bagacillo direct to the mud tank have been advocated as a solution of the problem.

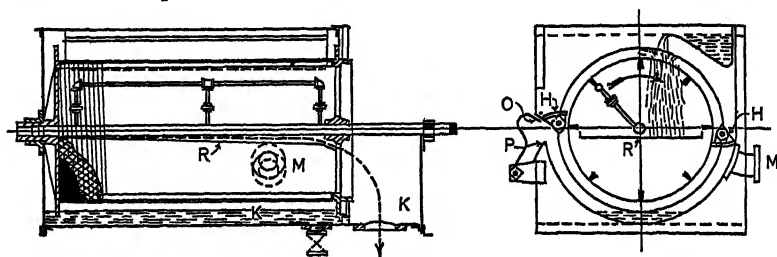


FIG. 14.—Peck Revolving Juice Strainer.

Other mechanical strainers used by some factories after the coarse straining are the "Hummer" and "Mitchell" screens of the type used in screening refined sugar. These consist of inclined screening surfaces which are either mechanically or electrically vibrated to keep the screening surface clear. These screens and the Peck strainer require too much headroom to be used for direct flow of the juice by gravity from the mills.

A recent advance in strainer practice involves the use of the strainerless juice pump introduced by the Honolulu Iron Works. This pump (Fig. 15) was designed for use with sewage and will handle juice containing bagasse without difficulty. A centrifugal pump, of the type used for drainage and dredging purposes, is also being tried out in Cuba for the handling of unstrained juices. With the ability to pump unstrained juices the mechanical strainers described above can be placed at any convenient point and the raw juice containing the bagasse fiber pumped to them. At the same time the thin juices returned to the mills need not be strained at all, a distinct advantage toward better mill sanitation and the prevention of fermentation in rehandled juices.

An installation of strainerless juice pumps is illustrated in Fig. 16. Compound maceration is taken care of by having one end of a pump handle 3rd mill juice and the other end 4th mill juice. A Peck strainer is placed just above the mills to screen all the juice going to process, this location being chosen to permit the easy return of the strained material to the bagasse on the mills



(in the present instance, to the shredder). The juice for maceration purposes is spread on the bagasse by means of a patented distributor.

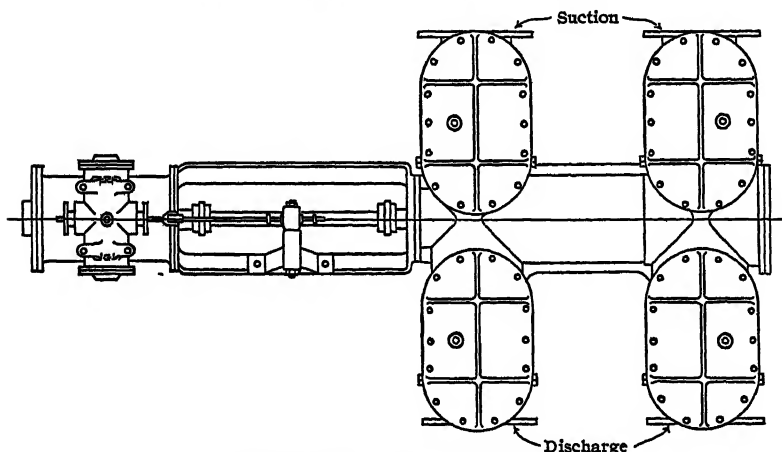


FIG. 15.—Strainerless Juice Pump.

In connection with mill sanitation it should be mentioned that all horizontal bolts, rivets and other projections in and around the juice stream and juice troughs are now recognized as sources of fermentation and potential sugar losses. A handful of juice-soaked bagasse caught on such a projection will sour very quickly under the warm conditions prevailing under mills and will be a

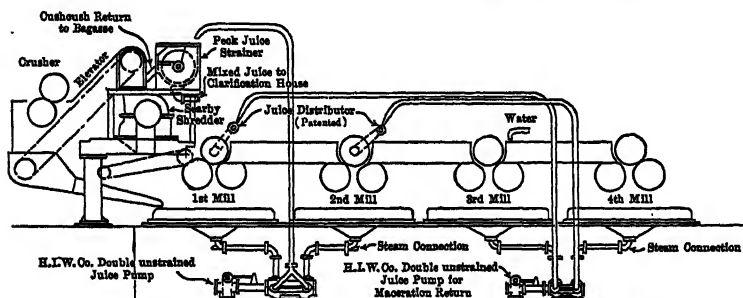


FIG. 16.—Installation of Strainerless Juice Pumps and Compound Saturation.

source of infection to the juice flowing over or past it. Mill designers are now seeking to avoid all obstructions to the free flow of the juice.

**30. By-product of Milling.**—The by-product of milling cane is bagasse or megasse (English colonies), the woody fiber of the cane with the residual juice and moisture derived from the saturation-water. This material supplies a very large part and in many instances all the fuel required by the factory.

Many attempts have been made to utilize this material in paper manufacture, but in general they have not been commercially successful. In Hawaii a low grade paper has been made from bagasse for several years which is used as a "mulch" to cover the ground between the cane plants to keep down the weeds, and more largely in the pineapple plantations for a similar purpose.

Havik<sup>10</sup> was commissioned by the Government of the Dutch East Indies to investigate the fibers of Java and the other islands with a view to paper manufacture. He obtained 32 per cent of the weight of dry bagasse in dry unbleached paper. The bagasse was from Cheribon cane. The frequently stated yield of 52 per cent of paper is disproved by Havik's experiments.

A successful use of bagasse fiber which has almost superseded its use as a fuel in Louisiana is in the manufacture of a building and insulating board sold under the trade name of "Celotex." The bagasse is baled as it comes from the mill and the bales are stored in the open in large piles covered by roofing paper. When taken to the manufacturing plant the bales are broken, and the bagasse fed through a shredder to a cooker which removes the resins, waxes and nectocellulose,<sup>11</sup> at the same time rendering the fiber tough and flexible. From the cooker and washers it goes through paper-mill beaters and refiners to separate the bundles of fibers. The waterproofing material is added at this point and the mixture of fiber and water is fed to the machine forming the board, which is formed by the process known in pulp-handling as felting. No adhesive is used, the strength of the board being due solely to the inter-weaving and entangling of the fiber. The wet board as it comes from the machine is fed to a continuous hot air dryer 800 feet long, the board emerging dry in sheets 4 feet wide and 950 feet long to be cut by saws into convenient sizes. The material is made in two thicknesses,  $\frac{7}{16}$  inch for building board and  $\frac{5}{16}$  inch for box material. Its manufacture in Australia is also being undertaken.

Investigations are now under way in several plants in Cuba looking toward special uses for the cellulose of the bagasse. This may be separated mechanically by sifting or other means and is believed to have particular value in lacquer and artificial silk manufacture. There is no question that with so many factories producing an excess of bagasse over fuel requirements, plans will be found for employing this material in ways which will make it a valuable by-product.

<sup>10</sup> Int. Sugar Jour., 14, 52.

<sup>11</sup> T. E. Monroe. La. Planter. July 15. 1922 (lxix).

## CHAPTER IV

### STEAM PLANT AND FUEL

**31. Steam Boilers.**—The boiler capacity required in a cane-sugar factory varies with the manufacturing equipment, the quality of the cane, the quantity of saturation-water and the grade of sugar produced. More boiler capacity is necessary for poor than for rich cane, and for the manufacture of plantation white sugar than for raw sugar. If the factory equipment includes multiple application of the vapors generated in evaporating the juice, in juice-heating and for other purposes, as is customary in the beet industry, its boiler requirements are lessened. It is customary to state the boiler capacity required by a factory in nominal or rated boiler horse-power per cane capacity-ton.

A large Cuban factory, having good equipment, including a quadruple-effect evaporator, but not using pre-evaporators or utilizing the vapors from the first or other pans of the multiple-effect evaporator in juice-heating or evaporating sirup, requires from 1.25 to 1.50 nominal boiler horse-power per 1 capacity-ton, or about 12.5 to 15 square feet boiler heating surface.

Hawaiian "standard practice" calls for 450 sq. ft. heating surface per ton of cane per hour (equal to 45 rated boiler horse-power to 24 tons per day) or 1.8 rated b.h.p. per ton capacity per day. Patrick Murray<sup>1</sup> reports boiler capacity in South Africa as ranging from 29 to 44.6 sq. ft. heating surface per ton per hour which equals from 1.16 to 1.79 b.h.p. per ton capacity per day. Good mill-work and the utilization of the vapors of the multiple-effect in juice-heating may materially reduce these numbers. The manufacture of white sugar with its attendant increase in steam-consumption for evaporating wash-waters, etc., may increase the steam requirements 15 per cent or more.

Both fire-tube and water-tube boilers are used in cane-sugar factories. Many Cuban factories select water-tube boilers on account of their greater safety. The fire-tube boiler is often considered to have an advantage over the other type on account of its large water capacity, which fits it to meet the very irregular demands of the factory for steam.

Recent tendencies in Cuba have been toward water-tube boilers in large units ranging from 600 to 1200 hp., the straight-tube types being more popular. In Hawaii water-tube and fire-tube boilers are about equally divided.

**32. Fuel.**—The usual fuel of the cane-sugar factory is bagasse, the residue from the milling of the cane itself. Bagasse contains a large percentage of moisture which varies with the grinding speed and the efficiency of the milling, the average for Cuba and Porto Rico being around 49 per cent. In Hawaii the

<sup>1</sup> South African Sugar Journal. 1924.

moisture content is frequently 40 per cent or even lower (average about 44 per cent) due to the much slower grinding rates and consequent higher efficiency of the milling. Many small mills of the older types in Louisiana turn out bagasse ranging above 55 per cent water. Where cane is of moderate cost, labor and fuel very dear and sugar sells for a low price, the factory may bring in larger net returns by grinding a large quantity of cane less efficiently. These conditions obtain more or less in Cuba where emphasis on capacity has been stressed at the expense of efficiency. This large grinding produces more fuel in proportion to radiating surfaces and by keeping the percentage of maceration water to a minimum the use of extra fuel may be appreciably reduced, but this is necessarily at the expense of sucrose extraction.

During the last few years the tendency in Cuba has been toward higher extractions through more efficient milling and the use of larger percentages of maceration water. At the same time many of the larger plants which are electrified have given such close attention to fuel economies that the problem of taking care of an excess of bagasse has presented itself, although 25 per cent or more maceration water is being used. The majority of factories still resort to the use of extra fuel, generally oil or wood, which constitutes an appreciable addition to the cost of manufacture.

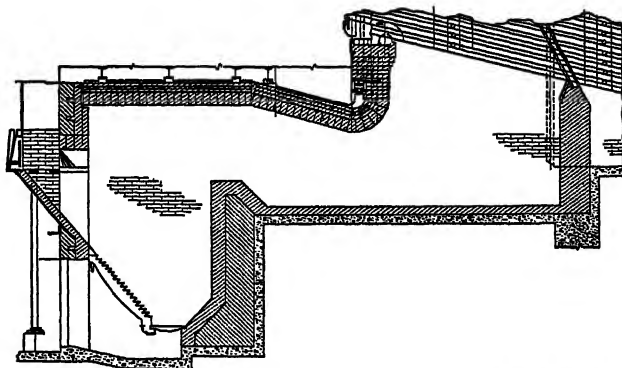
The partial drying of the bagasse by the waste heat in chimney gases to increase the net fuel value has been advocated by many and is actually practiced in a few small plants in some European colonies. It offers many mechanical difficulties, together with considerable danger of fire in the driers. It can be shown that preheating the air admitted to the furnace for combustion purposes will effect as great a saving as drying the bagasse<sup>1</sup> with much simpler equipment. The Ljungstrom preheater has recently been installed at "Central Cuba" in Cuba where it is said to save 15 per cent of fuel. Similar installations are in use in Mexico and South Africa, with 20 per cent saving reported from the latter country.

**33. Bagasse Furnaces.**—The first furnace for the burning of green bagasse for generating steam was patented by Samuel Fiske, the inventor of the cane-shredder. It was first used in Louisiana and then in Cuba at "Soledad" Cienfuegos. This furnace consists of an oven fitted with horizontal grate-bars upon which the bagasse is burned. A single furnace was often connected by means of flues with several boilers, though preferably in entirely new installations with but two. In the latter case the furnace was under the front end of the boilers. Forced-draft was used in the early installations, since the bagasse often left the mills with as high as 60 per cent moisture. Almost simultaneously with Fiske, Frederick Cook introduced his green bagasse-burner into Cuba at "Hormiguero," also after using it in Louisiana. In this type of furnace the bagasse is burned on a hearth in an oven placed between two water-tube boilers. Air is forced into the burning bagasse through tuyeres. Prior to the introduction of the Fiske and Cook burners all bagasse was sun-dried in Cuba before burning it. The inventions of Fiske and Cook had a profound influence upon sugar-manufacture through enabling, and, in fact, forcing the

<sup>1</sup> Report of Sub-Committee of Natal Sugar Technologists. The Planter, October 16, 1926.

factories to operate day and night instead of but fourteen hours, and in sending large numbers of people to the fields who were formerly employed in drying and firing bagasse. The step-grate furnace is the third and most recent type (see Fig. 17). In this type there is usually a furnace in front of each boiler. The grates are inclined and resemble a step-ladder, and in fact are often termed "step-ladder" grates. These grates are very long and narrow. The bagasse falls upon the top steps of the ladder and gradually works its way to the small flat grate at the bottom.

Modern installations of all three types of furnaces are very efficient. The flat grate and hearth types are generally used in Cuba and Porto Rico, while the step grates are favored in those countries where the moisture content of the bagasse is lower (Hawaiian Islands, the Philippines and Java). Forced draft, which as stated before, was used in the earlier installations was later abandoned until its use was almost entirely discontinued. In the more recent



*Hedges, Walsh & Widener.*

FIG. 17.—Step Grate Bagasse Furnace.

furnace installations, however, forced draft has been generally readopted and with its use ratings as high as 200 per cent of the rated boiler horsepower have been obtained while constant operation at 175 per cent rating is not at all unusual.

The problem of furnace design for bagasse has been given much study lately and it is now recognized that the essential factor in burning such a wet fuel is that as high a furnace temperature as possible be maintained. In burning such dry fuels as coal or oil the boilers are placed immediately above the fires to absorb as much heat as possible and reduce furnace temperatures in order to protect the brick settings, whereas with bagasse the opposite is true. Combustion must be completed in a separate compartment before the gases strike the cool surface of the boiler, otherwise temperature may be lowered to a point where losses will occur due to carbon monoxide or unconsumed carbon. According to Renton<sup>3</sup> the highest temperature attainable with 50 per cent moisture and 100 per cent excess air is 1985° F.

<sup>3</sup> *The Planter*. May 15, 1926.

The most recent furnaces have flat tops (so-called "flat arches") which besides having many advantages of construction, distribute the hot gases to the boiler more evenly than the circular brick arch formerly used. (See Figs. 17 and 18.) Another important detail in most recent designs is the "mixing wall," above the bridge wall (Fig. 17) which directs the gases at the top of the furnace downward to promote mixing and prevent stratification of the gases, thereby aiding in securing complete combustion. The use of forced draft also aids in mixing the gases and avoiding stratification. The downward slope of the roof reflects the heat back on the fuel bed, promoting the drying of the incoming bagasse.

**34. Fuel Value of Bagasse.**—The fuel value of dry bagasse shows great uniformity throughout the world. Numerous calorimeter combustion tests in Cuba, Louisiana, Hawaii, Natal and other sugar countries show that bagasse

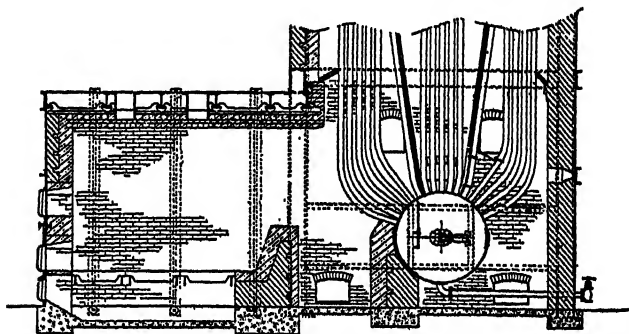


FIG. 18.—Flat Grate Bagasse Furnace.

perfectly free of moisture contains between 8300 and 8400 heat units (B.t.u.). Dr. R. S. Norris<sup>4</sup> found a value of 8100 in Hawaii but later investigators there corroborate the figures given above (8350). In considering the actual fuel value of bagasse as it is burned upon the grates it must be taken into account that a certain number of heat units are absorbed in evaporating its moisture and heating the resultant vapor to the temperature of the chimney. Further, a part of the fuel is consumed in heating the excess air which is drawn through the grates, as well as in heating the products of combustion, this heat loss being dependent upon the temperature of the flue gases passing up the stack.

Below is given a table showing the actual number of B.t.u.'s per pound of bagasse, with varying moisture content and different percentages of excess air.

Heat value per pound of dry bagasse, 8350 B.t.u.

Bagasse assumed to have the following composition:

$$C = 46.5; H_2 = 6.5; O_2 = 46.0$$

<sup>4</sup> Bul. 40 Hawaiian Sugar Planter's Exp. Station.

HEAT VALUE PER POUND OF BAGASSE BURNED  
(Stack Temperature, 500° F.)

Per Cent Moisture in Bagasse	Theoretical Amount of Air	Per Cent Excess Air			
		50	100	150	200
40	3747	3572	3400	3225	3054
41	3664	3494	3321	3151	2985
42	3579	3412	3244	3377	2902
43	3494	3329	3167	3004	2840
44	3400	3249	3089	2928	2769
45	3326	3169	3012	2854	2697
46	3241	3087	2933	2778	2624
47	3160	3007	2856	2704	2553
48	3077	2928	2779	2631	2482
49	2993	2851	2700	2557	2409
50	2910	2767	2624	2481	2338
51	2829	2689	2548	2416	2275
52	2744	2607	2469	2332	2194
53	2664	2528	2393	2259	2124
54	2582	2450	2319	2188	2057

These figures represent the actual number of B.t.u.'s available at the burners when conditions as regard excess air, stack temperature and moisture are as indicated.

The amount of excess air actually being used may be arrived at by determining the  $\text{CO}_2$  in the flue gases (Chap. XXXVIII) and using the curve given in Fig. 136 showing the relationship between  $\text{CO}_2$  by volume and percentage of excess air. One hundred per cent excess air is generally taken as good average practice.

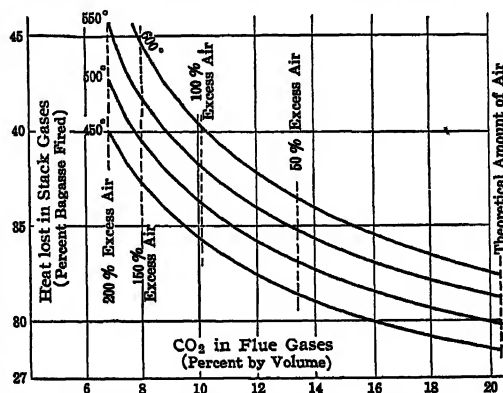


FIG. 19.—Heat Lost in Stack Gases.

The effect of variations in stack temperatures on fuel losses is shown in the chart (Fig. 19) for a bagasse of 49 per cent. It will be noted that approximately 6 per cent more bagasse will be burned with a stack temperature of 600°

F. than with one 450°, using 100 per cent excess air, all other conditions being equal.

For comparative purposes, one ton of bagasse under average Cuban conditions (49 per cent moisture, 100 per cent excess air) closely approximates the fuel value of one barrel (335 lbs.) of crude oil and of one-quarter of a ton of coal, using the same firing conditions.

**35. Molasses as Fuel.**—According to Norris (*loc. cit.*) Hawaiian final molasses has a somewhat higher thermal value than green bagasse. It will be seen from these comparisons that molasses as a fuel is only economical where molasses prices are very low or other fuels abnormally high. When molasses is burned it is best applied by atomizing it with steam as in burning oil. In Java the molasses is sprayed over the bagasse as it leaves the last mill, thick molasses being heated to facilitate spraying. According to Van Harreveld <sup>5</sup> the molasses must be thoroughly absorbed by the bagasse to avoid excessive trouble with slag.

<sup>5</sup> Proc. Second Conf. Int. Soc. Sugar Cane Technologists. Havana, 1927.



## CHAPTER V

### AN OUTLINE OF THE CUSTOMARY METHOD OF MANUFACTURING RAW CANE-SUGAR

**Introductory.**—The purpose of this outline is to minimize the repetitions in future descriptions and especially to define many of the technical terms, at the same time illustrating them with examples. Terms used elsewhere than in the United States are included in parenthesis. The processes are described in full in a subsequent part of the book.

**36. Outline of the Manufacture.**—*Purification of the Juice.*—The defecation or clarification of the juice is accomplished by means of lime and heat. The acidity of the juice is neutralized by milk of lime and its temperature is then raised to the boiling-point. Practically all factories using the defecation process settle the juice in special tanks after heating, and decant the clear liquid or "clarified juice" from the "mud," "scum" or "slops" (Cuba = *cachaza*). The mud is filter-pressed and the filtrate or "press-juice" is mixed with the clarified juice. The precipitates retained in the press, "filter-press cake," are discarded or used in fertilizing the fields.

*Evaporation.*—The clarified juice is evaporated in multiple-effect vacuum apparatus to a "sirup" (Cuba = *meladura*; <sup>1</sup> beet industry = thick juice), containing approximately 45 per cent of water.

*Crystallization.*—The crystallization is accomplished in single-effect vacuum-pans under reduced pressure. The sirup is evaporated until saturated with sugar. At this point crystals or "grains" separate in the boiling mass. The pan is "charged" with sirup from time to time, as the water evaporates, and the sugar it contains is largely deposited upon the grain present without the formation of additional crystals.

The growth of the crystals is so regulated that they are of sufficient size when the pan has been filled at which point the mixture of crystals and sirup is concentrated to a dense mass, "massecuite," (beet industry = fill-mass) and the "strike" is then discharged from the pan. This latter is often termed a "strike" pan.

*Centrifugaling; Purging; Reboiling Molasses.*—The massecuite is conveyed into a mixer and from this is drawn into centrifugal-machines, "centrifugals." These machines have cylindrical perforated metal "baskets," lined with wire-cloth and perforated bronze-sheets and are spun at high velocity. The sugar-crystals are retained by the lining and may be washed upon it with water if desired. The mother liquor, "molasses," passes through the lining by reason of the centrifugal force exerted. The machine is stopped after the removal

<sup>1</sup> Pronounce "May-lah-dew-rah."

of the molasses and the sugar is "cut down," leaving the centrifugal ready for another charge of massecuite.

The molasses is returned to the vacuum pan for reboiling with a portion of high-grade sirup to obtain a second crop of crystals and this "second massecuite" is centrifugaled as before, yielding a sugar which is about the same quality as that from the first massecuite but a "second molasses" of much lower purity. When successive re-boilings have reduced the molasses to such a low purity that it will no longer yield any more sugar by crystallization, it is sold as "final molasses" or "black strap." The modern factory produces only one grade of raw sugar and final molasses. The development of the production of high-grade sugar from all massecuites of whatever purity has greatly complicated the pan-boiling systems.

**37. Factory Design and Construction.**—A large Cuban factory constructed in 1912 is shown in the frontispiece. The capacity has been greatly increased since it was built by adding an additional mill house (at the rear in this view) and extending the boiling house, the largest building in the group. This factory has three trains of mills and is the only plant in the world ever to produce a million bags (325 lbs. each) of raw sugar in one season. The buildings are of steel and the machinery is largely electrified.

## CHAPTER VI

### PURIFICATION OF THE JUICE

**38. General Considerations.**—The juice leaving the mills is mechanically strained as described on page 30 after which it is customary in most modern factories to weigh all the juice in tank scales (Sec. 372). The purification, which is termed "clarification," or "defecation," (though originally there was a distinction in the two terms), starts after the weighing station is passed.

The object of the clarification is to remove from the juice the maximum quantity of impurities at the earliest possible stage in the process. In raw sugar manufacture lime and heat are the agents almost exclusively used for this purpose. The manufacture of "direct consumption" sugars (*i.e.*, white sugar, "plantation granulated," "yellow clarified" or any other sugar intended for consumption without refining) involves the use of other chemicals in conjunction with lime. These processes will be considered later.

In the clarification by heat and lime the quantity of lime added, the method of adding it and the temperature to which the juice is heated all vary widely with differing conditions and in different localities, but in general sufficient lime is added to neutralize the organic acids present in the juice after which the temperature is raised to 200° F. or above. This lime and heat treatment forms a heavy precipitate of complex composition, part of which is lighter and part heavier than the juice, which contains insoluble lime salts, coagulated albumin and varying proportions of the fat, wax and the gums. The precipitate is flocculent and carries with it most of the finely suspended material of the juice which has escaped mechanical screening. The separation of this precipitate from the surrounding juice is almost universally accomplished by subsidence and decantation, the different forms of defecators and clarifiers to be described later being designed to carry out this separation as completely and rapidly as possible.

There has been increasing recognition in recent years that the degree of clarification has great bearing on the subsequent stations of the factory, affecting the pan boiling, the centrifuging, the quality of the products, and most important of all, the yield of raw sugar. The filtration speed in the refinery is also affected to a marked degree by raw sugar qualities traceable to the clarification. (See p. 143.)

**39. Methods of Adding the Lime.**—The lime is generally added in the form of a milk to the cold juice just after it leaves the weighing tanks and before it enters the heaters. In all factories of any size lime-circulating systems are in use; the lime being made up to the desired density in a tank as a suspension in water and then circulated to the various points in the plant where lime is

used, the excess returning to the supply tank. Some experiments were made in Hawaii about 1921 on adding the lime at the mills with a view to preventing inversion in and around the mills, juice strainers, etc., but results were not considered favorable. This practice is objectionable from a chemical standpoint as well as for other reasons because of the solvent action of lime on some of the constituents of bagasse fiber.

There are many who advocate adding the lime to the juice after it has been heated, generally to a temperature well above the boiling point. Geerlign<sup>1</sup> advises liming after heating in cases where the gum-forming *leuconostoc mesenteroides* is troublesome, as this organism cannot develop in acid juice. Maurice Bird<sup>2</sup> published results showing that "crude gums" were much lower in juices heated to 238° F. and then limed than in those heated to 212° F. after liming. The Gilchrist process (Sec. 49) employs a sucrate of lime added after the juice has been heated to 235° F. Agreement seems to be general that the gummy materials (reversible colloids, p. 49) are flocculated by heat in an acid medium, while they pass through the ordinary alkaline defecation with cold liming. Some observers claim that raw juice can safely be heated to high temperatures for a short time without fear of inversion, but the point does not appear to be well established. An authoritative study of liming after heating and liming before heating seems to be needed.

The addition of the lime may be either intermittent or continuous. In the older systems using open tank defecators without heaters the lime is added directly to each defecator as it is filling with juice. Where closed heaters are used, as in all modern systems, the juice is generally limed in the cold in a series of large tanks. At least three conical-bottomed tanks should be used, that each may be thoroughly cleaned after each filling. The lime precipitates are kept in suspension by rotating paddles or by a current of air forced through a perforated pipe. The latter is the preferable method, as the pipes interfere but little in cleaning the tanks. This system of adding the lime has the advantage of simplicity and permits of exact regulation of each tankful to any desired reaction, since there is usually time for such regulation after the tank has been filled.

A prompt and intimate mixture of the lime and the juice is highly desirable in order to avoid local over-liming. To effect this, Meeker of Central Tinguaro, Cuba, devised a simple arrangement by which the outlet valve of the lime measuring tank is actuated by the stream of juice flowing into the large liming tank, so that the lime is added in a small stream and intimately mixed with the juice as the latter enters the liming tank rather than adding it in one large dose as is frequently done in intermittent liming.

Continuous liming systems add the milk of lime to the juice stream in a constant stream, generally as the juice enters the heater pump. Continuous liming devices of special type have been identified with various patented defecating systems, but obviously any method of adding the lime may be used with any system of clarification. The Fleener continuous liming device has

<sup>1</sup> "Cane Sugar Manufacture," p. 144, 2nd Ed., London, 1924.

<sup>2</sup> Facts About Sugar. 23 (1923), 6. Louisiana Planter. 72 (1924), 13.

been favorably reported upon in Hawaii.<sup>3</sup> It consists essentially of a weir-box in the usual lime circulating system which delivers the milk of lime to a distributing pan beneath the weir. The distributing pan is divided into two parts by a plate, one part delivering the milk of lime to the juice in the centrifugal heater pump, while the other part returns the excess lime to the circulating system. By moving the weir-box longitudinally above the distributing pan more or less milk of lime falls in the part of the pan delivering to the juice. As is usual in continuous liming systems, a small pipe returns a stream of the limed juice to the liming device so that the operator may test the reaction.

An automatic electric method of adding the lime is being experimented with which will be described in the next section.

**40. Regulating the Quantity of Lime.**—The addition of the correct amount of lime is essential to good clarification. Too little lime will give poor settling, and a resultant cloudy juice, as well as possible losses by inversion, whereas too much lime will cause darkening of the juices, increase in the gummy substances (see p. 49), increased ash due to dissolved lime salts, and high molasses output.

The earliest method of determining the correct quantity of lime was by judging the appearance of the juice and the suspended matter in a test tube after liming and heating. The particles of suspended matter should move rapidly toward the surface at the sides of the tube and descend promptly at the center forming a compact cone. This method is still used by many defecator men, and all other methods are, in a sense, supplementary to this since the ultimate test of the correctness of liming is the rapidity and sharpness of the separation of the precipitate, together with the brilliancy of the clarified juice, commensurate with avoidance of excess lime salts in solution.

The use of sensitive litmus papers, turmeric paper, and titrations with calcium succrate solutions, until the recent adoption of hydrogen-ion concentration methods, were generally depended on, reactions being expressed as cubic centimeters of N/10 NaOH required to bring 10 cc. of the juice to neutrality to phenolphthalein. The use of litmus requires daylight, or "artificial daylight" from any of the several lamps giving light of this character. A juice limed to neutrality to litmus usually settles slowly. In raw sugar manufacture an alkaline clarification is generally practiced, though the accepted alkalinity varies from faint alkalinity to litmus, to neutrality to phenolphthalein as determined on the cold limed juice. In titration with standard alkali these would represent an acidity to phenolphthalein of from 0.0 to 0.50 cc. N/10 NaOH to neutralize 10 cc. of limed juice, while the corresponding pH values would be from about 7.5 to 8.5 on the cold limed juice. It is to be understood that there can be no definite relationship between reaction by titration and by pH. The pH gives the effective acidity and the titration the total acidity, and two juices of the same pH may differ quite widely in titrated acidity.

The introduction of hydrogen-ion concentration or pH control (see Chap. XXII) into raw sugar work has marked a distinct advance in the exactness of clarification practice. Any of the colorimetric methods are so simple as to

<sup>3</sup> H. G. Hall. *The Planter*, October 2, 1926.

permit of their use by ordinary skilled labor after the desired pH number has been decided upon and prescribed by the laboratory. It is of interest that the pH of the juice in all ripe normal canes is close to a fixed figure; about 5.10 (see page 14).

Paine and Balch <sup>4</sup> of the U. S. Carbohydrate Laboratory made a study in Porto Rico of the relationship between pH and defecation and found that for maximum clarification it was necessary to lime to between 8.0 and 9.0 pH on cold juice. They obtained fairly good clarification, at times, between 7.6 and 8.0 pH but never below 7.5 pH. They found that a drop of 0.2 to 0.3 pH occurs on heating juice to the boiling point, so this must be allowed for if tests are made after the limed juice has passed through the heater. These investigators showed that there is a steady fall in the pH of juice kept at the boiling point for six hours; juice that was originally limed to 8.0 pH dropping to 7.0 in three hours. <sup>5</sup> Inversion takes place to an appreciable degree at high temperatures at 6.8 pH, according to the findings of Walton, Hornberger and McCalip, <sup>6</sup> so it is advisable that the juice be limed to such a point that it will reach the sirup stage at not less than 6.8 pH.

In Hawaii the Experiment Station advises liming to 8.0-8.3 pH (tested after juice has passed through heater) which closely approximates neutrality to phenolphthalein. McAllep <sup>7</sup> reports that this cannot always be maintained because of the lack of press capacity and inability to take care of the increased quantity of settlings due to the high liming.

A study of the use of pH in Java clarification practice by Honig and Khainovsky <sup>8</sup> brought out that "it should be recommended that defecation in general should be so carried out that the clear juice has a pH of 7.0 to 7.5." They conclude, however, that it is not possible to state any definite pH for *all* juices, the criterion being that point at which as little mud and as much clear juice as possible is obtained. They showed that the extent of the drop in pH which occurs on heating the juice is a function of the amount of  $P_2O_5$  present; juices of high phosphate content showing a much more marked difference in pH before and after heating than those deficient in phosphate.

All the recent investigators on the relationship of pH and defecation seem to agree that liming the cold juice between 8.0 and 8.5 pH marks the optimum results for the following considerations: brilliancy of juice, volume of settlings, increase in purity between raw and defecated juice, freedom from destruction of glucose on the one hand and avoidance of subsequent inversion in the process on the other.

<sup>4</sup> The Planter. Feb. 12, Feb. 19, 1927 (LXXVIII).

<sup>5</sup> Much confusion has arisen in reporting clarification results and methods with pH numbers due to the failure to state definitely just where the pH is taken. As an example—a limed juice which shows 8.3 pH when cold would be about 8.0 after passing through the heater and 7.5 or even lower as defecated juice, depending on how much time it took to pass through the defecating system. For this reason such expressions as "liming to 7.5 pH" are meaningless unless the point at which the pH determination is made is stated.

<sup>6</sup> Ind. Eng. Chem. 17, 51. 1925.

<sup>7</sup> Proc. 2nd Conf. Int. Soc. Cane Tech. Havana, 1927.

<sup>8</sup> Int. Sug. Jour. Vol. 30 (1928), p. 264.

A most important point on which there is divergence of opinion is in the quantity of dissolved lime salts in the defecated juice in relation to the pH. Bond<sup>9</sup> says, "The remarkable fact shown is that, though lime is actually added to the juice, its CaO content may actually decrease up to a reaction of about pH 8 in the cold. Above this point the CaO content increases rapidly. Thus it is entirely possible to clarify juices with lime and obtain a clear juice of lower CaO content than originally present." Against this King<sup>10</sup> found that "at no reaction above 7.6 was there less CaO than that originally present." The work of Paine, Keane and McCalip<sup>10</sup> showed that increase in the quantity of lime (and consequently the pH) increases the lime-salts content of the clarified juice, and they warn against the detrimental action (long known to practical sugar men) of these excess lime salts.

It seems certain, that in attempting to gain certain advantages due to liming to higher alkalinities (rapidity of settling, clarity of juice, elimination of irreversible colloids, etc.) greater disadvantages may be encountered through the excess soluble lime-salts and the resolution of reversible colloids, or gummy substances.<sup>11</sup> A careful balancing of these and other considerations will influence the pH to which the individual factory finds it advisable to lime. Defecator and press capacity must be considered; the character of the juice is also a determining factor and in the case of juice from burned or deteriorated canes it is advisable to neutralize excess acidity with soda ash before adding the lime.

*Electric Recording and Control of Liming.*<sup>11a</sup>—"Automatic control of the liming of cane juice in the manufacture of raw sugar, operating on electro-metric principles, was tested in a Porto Rican central over a period of several months.

"The equipment consists of (1) a recording potentiometer fitted with a controller time switch, through relays, which causes the motor of the liming device to operate in the required direction at definite intervals; (2) tungsten and calomel electrodes fitted in a continuous flow juice chamber; (3) a temperature compensator which automatically corrected for the change in pH due to temperature variations; and (4) a tilting weir box (or other device for regulating the flow of lime) for the liming device.

"The electrode chamber is shown in Fig. 20. It should be of glass, as this is easier to keep clean from mold growth, sedimentation, fermentation, etc., and the inspection is made easier and more positive. It will be noticed in the sketch that two electrodes are used in parallel, this being for the purpose of insuring a positive control that might be unreliable in case only one electrode were used, and the tungsten filament should break. The electrodes need not be changed more frequently than once every forty-eight hours.

"The controlling mechanism is essentially an intermittent time switch operated by a rotating cam which was actuated through a system of gears

<sup>9</sup> The Planter, 74 (1925), 21.

<sup>10a</sup> The Planter. 79 (1927), 15.

<sup>10</sup> Ind. Eng. Chem. 20 (1928), 3.

<sup>11</sup> Paine, Keane and McCalip, *loc. cit.*

<sup>11a</sup> Balch and Paine. Ind. & Eng. Chem., Vol. 20 (1928), No. 4.

from the recorder motor. During the control period the time switch caused one of the two relays to close and this in turn closed the circuit to the motor operating the liming device. The direction and the amount the adjustment of the liming device was changed depended upon the balance of the potentiometer at the moment the control switch made contact; that is to say, whether at that particular moment, the juice required more or less lime, as indicated on the chart by the deviation of the tracing from the position of the control point setting.

"The cam and the time switch were so constructed that they permitted the recording of the pH of the juice during two-thirds of the period and the control of the pH only during the remainder of the time. By changing the

gear ratio the control could be made to operate at every three-, six-, or nine-minute interval. Under the conditions of the investigation the three- and six-minute intervals gave the best results in automatically controlling the addition of the lime to the juice."

41. *Temperatures.*—The temperature to which juices are heated in clarification varies widely, 194° F. (90° C.) and 238° F. (115° C.) being the outside limits. General practice is to heat to the boiling point or slightly above. Superheating (that is, heating well above the boiling point) gives more rapid settling and a compact mud but Balch and Paine<sup>12</sup> found that the clarified juice was darker and more turbid when heated to 110° C. than the same juice heated to 100°. Geerligs<sup>13</sup> finds no difference in the juices due to the higher degree of heating, while others claim distinct advantages.<sup>14</sup> These diverse opinions may be due to differences in the various juices tested, and possibly

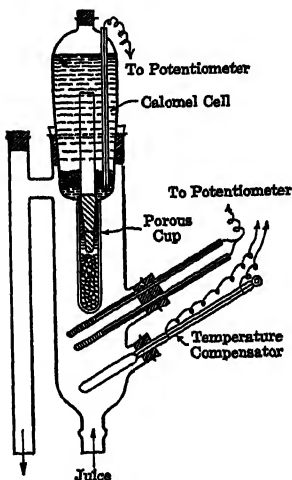


FIG. 20.—Electrode Chamber for Automatic Liming.

to the fact that this factor of clarification has not had the thorough study to which other phases have been subjected.

42. *Nature of the Precipitate.*—There has been an accumulation of evidence that the phosphate content of the raw juice is the major factor in efficient clarification. Walker<sup>15</sup> placed the minimum requirement at .02 gram  $P_2O_5$  per ml. of juice while McAllep and Bomonti<sup>16</sup> found that .030 to .035 gram was the lowest limit for good clarification. Bond<sup>17</sup> in a study of the nature of defecation also showed that phosphates are the determining factor. R. H.

<sup>12</sup> Ind. Eng. Chem. 20 (1928), 266.

<sup>13</sup> "Cane Sugar and Its Manufacture." 2nd Ed., London, 1924.

<sup>14</sup> Bird. The Planter, 61 (1922). Facts About Sugar. 23 (1928), 6.

<sup>15</sup> Ind. Eng. Chem. Vol. 15 (1923), p. 164.

<sup>16</sup> Hawaiian Sugar Planters' Record. 26 (1922), 139.

<sup>17</sup> The Planter. Vol. 74 (1925), No. 21.



King<sup>18</sup> agrees that they are the major influence but states that the ferric, aluminum and silicate ions present in the juice are precipitated when lime is added, forming a very gelatinous precipitate which occludes considerable organic matter. Paine, Keane and McCalip,<sup>19</sup> working on Porto Rican juices, corroborated the importance of the phosphate content, and showed that it was immaterial whether the phosphate was naturally present or added. The elimination of colloids was found to be a direct function of the percentage of  $P_2O_5$  present when the lime is added to a given pH.

An interesting possibility for the increase of the phosphate content of the juice was shown by Walker<sup>20</sup> by adding phosphate combinations to the soil. These experiments were substantiated to some extent by the Experiment Station of Porto Rico. If these experiments are borne out by subsequent work it will mean that a double value can be obtained through the use of phosphate if added to the soil, first by the promotion of growth and yield in the cane and secondly by improving the clarification qualities of juice from the cane.

Noël Deerr<sup>21</sup> showed that 75 per cent of the total non-sugars eliminated in the heat and lime treatment can be removed by filtration through asbestos (mostly bagacillo). Twenty per cent of the precipitate, according to Paine and Balch,<sup>22</sup> is ash which Bond has<sup>23</sup> shown to consist principally of  $CaO$  and  $P_2O_5$  with appreciable quantities of magnesium, aluminum, and iron. By calculation, therefore, organic matter, other than that originally present in the juice in suspension, is only 5 per cent of the total non-sugars eliminated; which must consist of insoluble salts of organic acids and colloids adsorbed by the flocculent phosphate precipitate.

Summed up, all these investigations indicate that defecation is essentially an inorganic reaction or combination of reactions in which the flocculent precipitate of calcium phosphate and other insoluble salts carries down with it the coarse suspended matter, occluding the finer suspensions and adsorbing some of the colloids. Paine and Balch<sup>24</sup> state that "within the range of moderate alkalinity used for cane juice defecation it seems certain that few if any of the reactions are quantitative."

The colloids eliminated in the process have been shown by Paine and his colleagues to be largely the irreversible type, i.e., those colloids generally high in ash which after drying will not redissolve or redisperse when taken up with water again. The reversible colloids (which consist of the gummy substances low in ash), pass through the defecation process almost untouched. They also showed that with an increase of lime salts in the defecated juice the amount of these reversible colloids or gummy substances increases in an almost direct linear relation, the assumption being that the excess lime over that needed for defecation either converts some of the irreversible colloids into the rever-

<sup>18</sup> The Planter. 79 (1927), 287.

<sup>19</sup> Ind. Eng. Chem. 20 (1928), 262.

<sup>20</sup> Ind. Eng. Chem. 15 (1923), 164.

<sup>21</sup> "Cane Sugar." London, 1920.

<sup>22</sup> The Planter. 78 (1927), No. 8.

<sup>23</sup> Loc. cit.

<sup>24</sup> Loc. cit.

sible type; or that it has a peptizing effect on the pectinous materials of the juice, or that it enters into direct chemical combination with the reversible colloids.

Excessive lime in defecation has long been known to give poor working low grade materials in the boiling house and dark-colored, gummy sugars. The work cited above offers a scientific argument in favor of avoiding the use of more lime than the minimum needed for good clarification.

#### APPARATUS AND PROCESSES

**43. Defecation and Clarification with Open Tanks.**—This is the process that was practised since the early days of the cane-sugar industry, with little improvement until comparatively recent years. It is now little used except in the smallest factories and is described here for its historic interest.

The raw juice is pumped from the mill-tanks to double-bottomed vessels termed "defecators." The inner bottom of the defecator is of copper and the outer shell of iron. As soon as the bottom of the defecator is covered with juice, milk of lime is added to it to neutralize the acids and steam is turned into the space between the bottoms. The workman regulates the steam-pressure so that by the time the defecator is filled with juice the latter will have nearly reached its boiling-point. The moment the thick scum that covers the surface "cracks" the steam is shut off and the juice is left undisturbed for the subsidence of impurities.

If the quantity of lime required has been properly gauged, a part of the impurities rise to the surface with the scum, forming the "blanket," and a part settle to the bottom of the defecator.

Several defecators are usually arranged in a series and are filled with juice in regular order. Allowing sufficient time after the "cracking" for settling, the mud is drawn off from the bottom of the defecator and sent to the filter-presses, the clear juice is run into the evaporator charge-tanks or into clarifiers, and finally the scum is sent to the presses.

In addition to the defecation process as already described, clarifiers or eliminators are frequently used. The ordinary clarifier is a tank fitted with steam-coils and a mud-gutter. The clear juice is drawn from the defecators into the clarifier, where it is boiled and skimmed and is then run into settling-tanks.

**44. Defecation Using Closed Heaters and Open Settlers.**—This is a modification of the old defecation process (43) adapted to large factories. A large proportion of the raw sugar made today is from this type of clarification system.

The juice is first limed in the cold in a series of large liming tanks as previously described and thence through closed heaters containing a large number of copper tubes. Approximately 1 square foot of heating surface per 1 cane capacity-ton is required in the heaters. Exhaust-steam is used in these heaters. The temperature of the juice is generally raised to the boiling point or a little above.

The heaters deliver the juice into large settling-tanks generally provided

with heating-coils. Sometimes the juice is simply settled and drawn off from the mud but experience indicates that much better results are obtained by the use of coils to reheat the juice to the "cracking-point" before settling.

The actual average working-depth of the tanks should not exceed 6 feet. The greater the depth of the liquid the longer the time required for the settling of the precipitates. The bottom of the tank should slope sharply from the rear end toward the front and from the sides to the center. The slope from rear to front should be at least 1 inch per foot, and preferably greater. This incline is necessary to facilitate the removal of the precipitates (mud, scum or cachaza). The juice and steam should enter the tank at the rear. The clear juice and mud should be drawn off at the front. The juice entering the tank at the rear tends to carry the mud forward toward the outlet. The coils should be of copper and about 4 inches in diameter and should provide approximately 25 square feet of heating surface per 1000 U. S. gallons of actual juice capacity in the tank (30-35 sq. ft. per ton cane per hour). The coils should be well above the bottom of the tank to facilitate the removal of the mud.

Dr. Spencer preferred a rectangular defecating tank, with rounded corners and the draw-down arrangement described below. These tanks may be of very large capacity. Many now in use have a working capacity in excess of 6000 gallons. While a deep tank requires a rather long time for settling, it has the advantage of yielding a small proportion of mud. In

so far as possible, it is preferable that the juice be conveyed from the defecators through pipe-lines rather than canals.

The draw-down arrangement is shown in Fig. 21. At A is a side outlet-elbow, with a ventilating pipe, F, leading above the top of the tank. In drawing down, the juice flows through the nipple, the elbow, A, and the cross valve, B, into the small collecting-box and thence to the trunk line, D. The angle-valve, C is opened for a moment before drawing down the juice, for the removal of mud that may have settled in the pipe-line or that may be near the outlet-nipple. The valves, B, C, are controlled from the working-platform H. The valves, G is used in washing the mud into the main line to the scum tanks, E. It will be noted that the pipe is ventilated to prevent syphoning and that the juice therefore stops flowing when its level reaches that of the bottom of the draw-down pipe and no mud can be drawn down. The elbow and nipple insure that juice shall be drawn from below the scum. It is advisable to locate

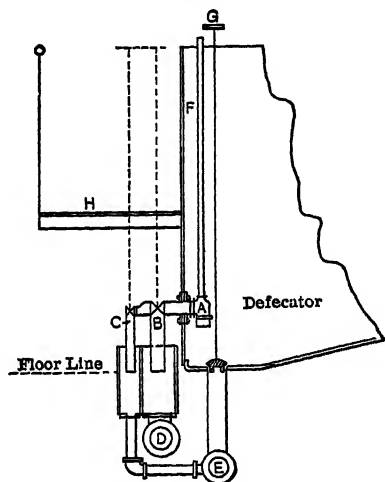


FIG. 21.—Draw-down Pipes for Defecators.

about three or four draw-down pipes in each defecator, with difference of level of about 4 inches, measured between centers. The lowest pipe should be about 12 inches above the bottom of the tank. The draw-down pipe should be about 3.5 inches in internal diameter in a defecator of 4000 gallons working capacity. If the juice has been properly limed and heated, there is no probability of drawing down mud, provided a sufficient settling time is allowed. Ample tank capacity and proper supervision of the liming are essential. Eight 5000-gallon tanks are sufficient for a grinding capacity of about 1400-1500 tons of cane per day (80 cu. ft. per ton cane per hour).

**45. Defecation Using Closed Heaters and Closed Settlers. Deming's Process.**—This process was invented by M. A. Scovell in a sorghum-sugar factory. The patents were purchased by Deming, who developed the process and made it available in practical work. This method came into prominence about twenty-five years ago but was largely superseded by intermittent settling (described in the previous paragraphs) until lately when other forms of continuous systems have come into use.

The juice is limed in the cold in Deming's process, in a single constant-flow tank. Milk of lime flows into the juice at the heater-pump intake and is thoroughly mixed with it in the pump and in transit to the heaters. The limed

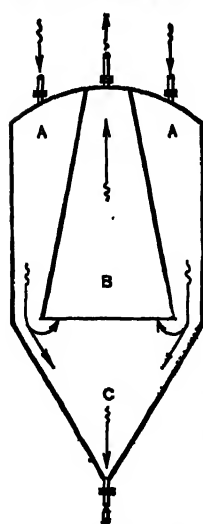


FIG. 22.—Deming Settling Tank.

juice is heated to approximately 235° F. and is then passed into an eliminator, where it parts with the gases, is slightly concentrated, and warms the incoming cold juice on its way to the heater. The eliminator is a cylindrical closed-iron-vessel with a conical bottom and is provided with a large heating-surface in copper tubes. Cold juice circulates through the tubes and condenses the steam set free when the hot juice enters the lower section of the eliminator. A partial vacuum is produced by this condensation and the air and other gases are withdrawn from the hot juice. The upper part of the eliminator is connected with a pump or the vacuum system.

The temperature of the juice should be reduced to about 210° F. in the eliminator. From here it is pumped into the outer compartment, A, of the closed settler or pressure-separator, shown diagrammatically in Fig. 22. The mud is drawn off continuously from the conical bottom of the tank at C and the clear juice is discharged from the central cone, B. In many factories using Deming's heaters and settlers, the juice is heated to only 212° to 218° F.

In his first experience with these tanks, the earliest of their type in Cuba, Dr. Spencer noted that the mud drawn off after several hours' operating had an offensive odor, indicating decomposition. He immediately installed very slowly moving scrapers in the tanks to prevent the mud from settling on the conical bottom of the separator and thus nearly eliminated decomposition. Certain spores resist the high temperature of the

heater and cause the decomposition. In Cuban practice it is found necessary to liquidate and clean the tanks as often as may be practicable. (See 85.)

**46. Cleaning the Heating-surfaces of Defecators and Juice Heaters.**—The heating-surfaces of coil-defecators used in combination with juice-heaters foul but little. Those in which the whole defecation process is conducted require frequent cleaning. Dilute muriatic acid is used.

Juice-heaters usually foul very quickly. The usual method of cleaning is to circulate hot caustic-soda solution through the tubes and follow this first with water and then with hot dilute muriatic acid. The soda solution may be returned to a storage-tank for repeated use, decanting it from the mud and adding caustic soda from time to time to maintain a strength of about 1 pound of soda to 7 gallons of solution. Occasionally the scale in the tubes is of a very resistant nature and must then be removed by scraping the surfaces. If the tube area is properly adjusted to the volume of the juice, so as to force a very rapid current, the scaling is much reduced.

**47. The Dorr Clarifier.**—This is a continuous system of defecation first introduced in Cuba about 1918 which has gained rapid favor throughout the various sugar-producing countries. The Dorr Clarifier (see Fig. 23) is used either for simple clarification—displacing defecators and mud tanks—or for double defecation in the operation of the Petree Process. The Dorr itself is a circular tank made of heavy steel plate, having an essentially flat conical bottom and a parallel conical top. Its interior is divided into a series of relatively shallow compartments by steel trays which are also conical and parallel to the top and bottom. These trays separate the compartments completely, excepting that there is a central opening extending through and from the cover through all the trays to the bottom, thus forming a mud passage which is common to all trays. Integral with the top of the tank and riveted to it, is a small tank or drum called the “feedwell.”

Above the clarifier tank body and supported by it is a steel truss. This truss is the support for the driving motor, the mud-thickening mechanism, and the diaphragm pump which withdraws the heavy mud. The mud-moving mechanism consists of a worm gear driving the vertical shaft which is suspended from the truss and passed through the feedwell and all the compartments to the bottom of the tank body. Keyed to this shaft, one just above the bottom of the tank, and one just above each of the trays, are spiders, the four branches of each of which support steel arms extending to the periphery of the tank. These steel arms carry flexible palettes of brass which rest lightly upon the trays and the bottom.

A small motor, supported on the superstructure, drives the worm gear which turns the shaft and causes the brass palettes to sweep over and in intimate contact with the floors of all compartments. In the feedwell, attached to and revolving with the motion of the shaft, are four radial paddles for pushing the foam into a discharge canal. Just beneath the cover of each of the trays, at uniformly spaced peripheral points, are the clear juice overflow pipes. In each compartment, considered as a settling unit, all these peripheral overflow points are connected to an internal header which passes through the side

wall of the tank body. There are as many take-out pipes for clean juice as there are compartments in a clarifier, generally four or five in number.

All the clear juice headers extending from the compartments are connected into a steel overflow box or juice collecting tank near the top of the apparatus. Each outlet pipe is provided with a sliding sleeve which renders adjustable the amount of juice overflow from each compartment. The force which causes the juice to flow through the clarifier is the difference in level between the sur-

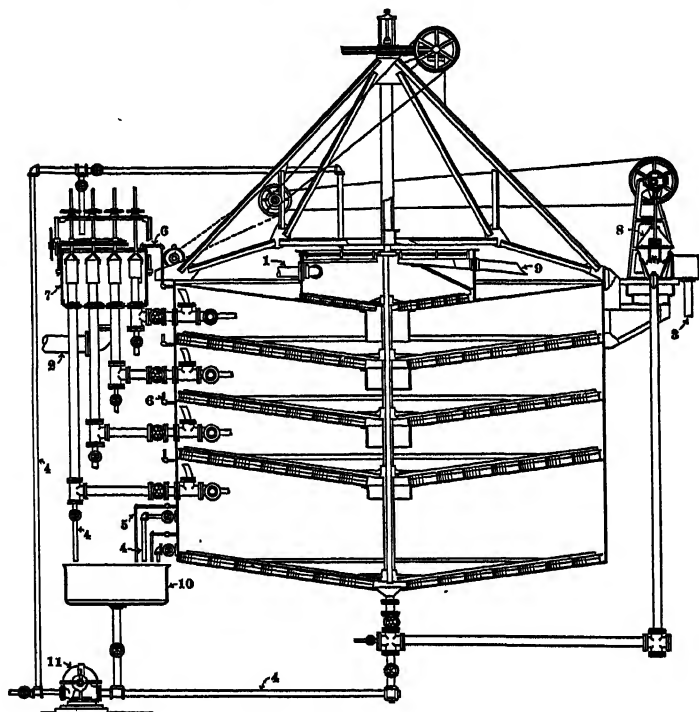


FIG. 23.—Dorr Clarifier.

face of the juice in the feedwell and that of the top of the adjustable sleeves in the overflow box.

The operation of the clarifier is thus continued indefinitely. Hot limed unclarified juice enters the feedwell, the foam rises and is removed, the heavy solids settle directly through the central opening to the bottom, the light bagacillo tends to travel toward the periphery with the juice, but the motion is so slow that this bagacillo settles upon the trays with a portion of the agglomerated solids. The revolving arms with their brass palettes pull this mud to the opening at the center of each tray and it falls to the bottom to form a part of

the layer of dense mud which accumulates there. The diaphragm pump withdraws this heavy mud as rapidly as it forms.

With most grades and conditions of cane, the mud withdrawn will carry only about 5 per cent of the total juice instead of the usual 10 or 20 per cent discharged from defecators. The difference in temperature between inlet and outlet lies between 2° and 4° F.

The advantages claimed for the Dorr are: Saving in labor, fuel and space, cleaner sugars and reduced filter-press capacity requirements, reduced losses in press cake and more efficient boiling house work.

**48. The Petree Process.**—The Petree Process consists of double defecation and the return of the resulting mud to the bagasse. The mill juices are separated into a high purity, high density "primary" juice and a low purity, low density "secondary" juice. These juices are treated separately. The "primary" juice, this term being used to denote the mixture of the limed crusher and first mill juice, together with the clarified "secondary" juice is pumped through the primary juice heater where it is brought to a temperature of 212° or higher, depending upon local practice, to the primary Dorr.

Under normal conditions about 95 per cent of the now clean and bright mixed juice leaves this primary Dorr at a temperature of 208–210° F. The quantity of juice, which is carried with the muds is therefore small. The second mill juice is limed separately and after being mixed with the rich settlings from the primary clarifier, is pumped through the secondary juice heater to the secondary Dorr.

The "secondary" mud, resulting from the settling of the mixture of the second mill juice and the primary mud, is usually diluted with the fourth mill juice, and this mixture is distributed uniformly and continuously over the bagasse as it issues from the second mill. The third mill juice is returned to the bagasse blanket behind the first mill. Local conditions determine the exact point or points at which the mixture is applied. (See flow sheet, Fig. 24.)

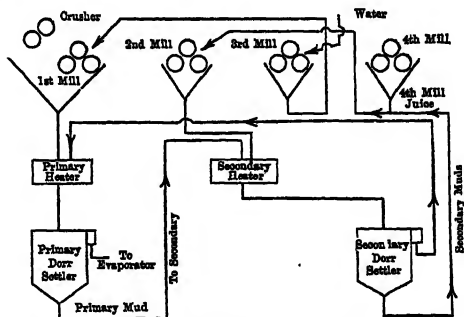


FIG. 24.—Flow Diagram of Petree Process.

The Petree Process makes use of a mechanical device to lime the juices as they come from the mills. This apparatus delivers milk of lime at a uniform rate into a spout. Across this spout is placed a baffle gate (in the nature of a weir), under which the lime milk flows in a thin uniform sheet. This sheet after passing the gate is divided into three parts by two adjustable splitters placed in the bottom of the spout. The three parts thus produced are received into the three compartments of a partitioned cast-iron box. From this box a part of the lime milk flows to the primary juice, a part to the secondary juice,

and the excess returns to the tank of the apparatus. The whole blanket of moving bagasse is automatically used to filter the very fine suspended solids from the weak juices of the last mills of the tandem. By disposing of the scums and settlings with the bagasse, the use of filter presses becomes unnecessary.

*Advantages of the Petree Process.*—First cost and repairs of filter-press cloth, maintenance of filter-press pumps, washing machines and other auxiliary equipment, plate and frame renewals and the cost of disposing of the press-cake. It is stated that in factories grinding up to 2000 tons of cane per day the use of the Petree Process accounts for the removal of about fifteen men from the payroll. The solid matter of the mud becomes available combustible, equal in value pound for pound to the bagasse, increasing the amount of the latter by about 5 per cent. The elimination of fiber, gums and waxes from the juices results in better heat transmission in evaporators and pans. The loss in presscake as such is eliminated. Users of the Petree Process claim an average saving of about 1 per cent on the weight of the sucrose in cane.

*Disadvantages of the Petree Process.*—Because of the return of settlings to the mills it is impossible to weigh the cold juice at the usual point and the chemical control of the factory is rendered much less positive. The earliest point in the process where the true control can begin is on the clarified juice as it enters the evaporators, and this necessitates the weighing (or measuring) of a very hot liquid, a difficult operation to carry out exactly. This radical change in the control methods makes a comparison between the figures in a factory employing the Petree process and one using ordinary single defecation of doubtful value. Furthermore, even admitting the accurate estimation of the clarified juice, the entire clarification process is omitted from the chemical control, unless unreliable inferential methods or equally unreliable cane sampling is resorted to. The "secondary" clarification of the thin juices at an acid reaction (6.4 pH) offers the possibility of inversion, but if such occurs the loss will not be shown by the technical figures. In spite of these objections advanced by many technologists the Petree Process is coming into increasing use each year.

**49. Gilchrist Process.**—This process, which has several distinctive features, is not yet in general use. The juice is first heated to 238° F. (114.5° C.) and is then sent through a "coagulator" which is essentially a tank with a stirring device into which is sprayed sucrate of lime regulated to give the desired reaction. Comment has already been made on the effect of heating before liming—precipitation of gums, colloidal silicates, etc., and a further advantage in flocculation of the precipitate is claimed through the very intimate mixture of the juice and the lime sucate in the form of a spray.

From the coagulator the limed juice goes to the "Gilchrist Rapid Settler" which is similar in general form to the Dorr but differs radically in that it has a "parallel-flow" for the clear juice and the settlings. The juice and settlings both move toward the outside of the trays, the mud being removed from the lower rim while the clear juice is drawn out of the top of the compartment. The manufacturers claim very rapid settling and clearer juice through the operation of this "parallel flow" principle.



50. Horne's "Super-defecation."<sup>25</sup>—This is a double-defecation process patented by W. D. Horne in which the first defecation is carried out by adding lime until no further precipitate is obtainable (strongly alkaline) and heating to 150° F. (66° C.) only, settling, and drawing off the clear juice by any of the usual methods. To the clarified juice is then added sodium phosphate (or acid calcium phosphate and sodium carbonate) to precipitate the excess lime salts, after which the double-defecated juice is again drawn off. The settlings from both defecations are combined and filter-pressed together. The juices from this process produce light-colored sugars low in ash and suspended matter and high in test.<sup>26</sup>

#### DEFECATION IN WHITE-SUGAR PROCESSES <sup>27</sup>

51. Remarks upon White-Sugar Processes.—Factories producing plantation white sugar, with few exceptions, make a "near" or "off" white product as compared with the American refiner's standard granulated sugar. In occasional "runs," however, in well-equipped factories, the product is almost or quite indistinguishable from refined sugar.

It is difficult to produce a perfectly uniform quality of sugar in the factory owing largely to the variable purity of the raw material. Judging from the opinions of many producers of white sugar, that made by the carbonation processes is of more uniform quality than by the exclusively sulphitation processes. The later are usually the cheaper processes.

There is a marked rise in the coefficient of purity of the juice in the carbonation process. This rise often exceeds two degrees. It is a true rise and is reflected in an increased yield of sugar. The rise of purity by sulphitation is not so great as by carbonation and de Haan claims that the latter process increases the yield of sugar more than 2 per cent over sulphitation processes and more than justifies the increased cost.

There is considerable difference of opinion as regards the possibility of making as white a product from the very dark-colored canes as from the light yellow and so-called white canes. The effect of the dark color is reduced by increase of lime in the defecation as in the carbonation and Bach processes. It is very probable that the best white sugars are produced from the light-colored canes.

Great attention to detail is essential to successful white-sugar manufacture. A little carelessness in the carbonation or sulphitation and filtration will result in a poor product. Double purging of the sugars in the centrifugals reduces the risk of staining the sugar through the necessarily imperfect removal of the molasses. Thorough cleanliness from start to finish is very essential; not only, from the point of view of color, but also of yield of sugar.

52. Sulphitation Process of Louisiana.—This is one of the simplest of the

<sup>25</sup> The Planter. Vol. 72 (1924), No. 11.

<sup>26</sup> Horne Ind. Eng. Chem. Vol. 16 (1924), 732, Vol. 20 (1928), 680.

<sup>27</sup> W. H. Th. Harloff's "Plantation White Sugar." 2nd Ed., London, 1920, should be consulted for a very thorough study of carbonation and sulphitation in the cane industry.

sulphitation processes and when skillfully conducted produces a very good, though irregular, quality of "near-white" or "off-white" sugar.

The cold raw-juice is pumped through a sulphur-tower or box, in opposite direction to and through a current of sulphurous acid gas. The juice should absorb as much of the gas as is possible (pH 3.4-3.8). This sulphitation is followed by liming to very slight acidity to sensitive litmus paper (pH 6.0-6.6) and the juice is then heated, settled and decanted, as is usual in the defecation process. The juice is usually reheated to boiling and then brushed before settling. Evaporation to sirup follows. Many factories boil and brush the sirup also. This brushing is a wasteful process, since it entails large consumption of fuel and loss of sucrose. Heating to the boiling-point is sufficient. The concentrated juice is also sometimes sulphited.

This is a very old process, and possibly originated in the English or French colonies. It is interesting to note that it has always been the custom to work the juice faintly acid to sensitive litmus to prevent coloration of the sugar. This condition corresponds to the essential one of the recent Java acid thin-juice process.

Many modifications and minor improvements of this process are in use in Louisiana factories: Double defecation using phosphate of soda in the second precipitation with subsequent bag filtering; bag filtration of the sirup after a resulphitation; pressure filtration of the sirup using a filter-aid (Filter-Cel) and the use of hydrosulphite of soda in the sirup and in the pans are among those expedients that have been adopted to give a higher grade of white sugar.

**53. Sulphitation after Liming.**—This process differs from the preceding in adding the lime, in large excess over that required to neutralize the juice, before sulphitation. Approximately 8 gallons of milk of lime of 26.5° Brix is used, thus producing a very heavy precipitate with the sulphurous acid and which may be readily removed by settling and decantation. If a larger quantity of lime is used, *e.g.*, 10 to 12 gallons, the precipitate may be removed by filter-pressing. The sulphitation is continued to neutrality to phenolphthalein. The decanted or filter-pressed juice, after concentration to sirup, is usually cooled and sulphited to slight acidity. An acidity equivalent to that required in 10 cc. of sirup to neutralize 25 to 30 cc. of 100th normal alkali is a suitable amount.

*Bach's Sulphitation Process.*—This process is extensively used in Java, under the patents of its inventor, N. B. Bach. Lime is added and is precipitated by sulphurous acid at two stages, each followed by subsidence and decantation or by filter-pressing. Very little if any more lime is used than in the process described in the preceding paragraph.

From 5 to 7 gallons of milk of lime of 26.5° Brix is added to each 1000 gallons of cold raw juice. This is then sulphited to neutrality to phenolphthalein and finally heated to full boiling, settled and the clear juice decanted as in the ordinary defecation. The clear juice is evaporated to the customary density, approximately 55° Brix.

The sirup obtained as above described is passed through a cooler and its temperature is reduced to about that of the factory's water supply. From 16 to 17 gallons of the milk of lime per 1000 gallons are now added to it and it

is then sulphited to neutrality to phenolphthalein, or the full quantity of sulphurous acid in solution may be added to the sirup prior to the lime. Since the volume of the sirup is about 30 per cent of that of the original juice, the total volume of milk of lime used per 1000 gallons of juice is from 10 to 12 gallons. Steam jets are used to beat down the foam during the sulphitation. The sulphited sirup is heated to about 194° F. (90° C.) and is filter-pressed. The press-cake is washed with water in the press and a large part of the sugar it contains is recovered. The filtration is rapid and the cakes are firm and well formed.

The filtered sirup contains some bisulphite of lime and is therefore heated to about 195° F., to decompose this and other bi-sulphites. The heated sirup is usually run into settling-tanks, and after the deposition of the precipitates the clear liquor is decanted and cooled as previously, and is then sulphited to distinct acid reaction to phenolphthalein and is then ready for the vacuum-pan (pH 6.1).<sup>28</sup>

Bach's process is sometimes slightly modified by separating all of the precipitates by decantation and mixing and filter-pressing them together.

This process requires approximately 0.055 per cent of sulphur on the weight of the cane. The sugars are of good quality.

## CARBONATION PROCESSES

**54. Preliminary Remarks.**—The ease with which cane-juices yield to the ordinary defecation and sulphitation processes has retarded an extension of the carbonation process, even in Java, where it has its largest application. The U. S. Department of Agriculture conducted extensive experiments on a manufacturing scale with carbonation of sorghum-juices in Kansas and cane-juice in Louisiana, over forty years ago. Dr. Spencer was active in this experimental work with Dr. H. W. Wiley, then Chief of the Bureau of Chemistry. As the Government reports show, these experiments were satisfactory from a manufacturing, but not financial point of view. They brought out the necessity of carbonation at temperatures well below 60° C., as is now practiced in Java, though this possibly originated with the French in the early Spanish installations.

There are two distinct carbonation processes, viz.: the single, in which all the added lime is saturated in one operation, and the double carbonation, in which the juice is treated twice with the gas. In the double process, a part of the lime is carbonated and removed by filtration and this is followed by a second carbonation in which the remaining lime, or this and a small additional quantity, are completely saturated with carbonic acid gas.

The single process has been materially and successfully modified by J. S. de Haan, thereby reducing the quantity of lime required and the expense of the process.

**55. Single Carbonation.**—A quantity of lime in the form of a milk and varying somewhat with the quality of the cane, is added to the cold raw juice.

<sup>28</sup> Farnell, *Int. Sug. Jour.* 27 (1924), 141.

This quantity of lime is approximately 1 per cent of quick-lime in terms of the weight of the cane. The facility with which the carbonated juice is filter-pressed is the principal factor in determining the quantity of lime which should be thoroughly slaked and efficiently strained through fine wire-sieves. The juice is usually limed in the carbonation-tank. The single carbonation is an intermittent process, *i.e.*, the tank is completely emptied after each operation.

The tanks for the single carbonation and the first carbonation in the double process in modern installations are very deep, often exceeding 22 feet in depth to take care of foaming. The tank is usually filled to less than one-fourth its depth. Pipes leading above the factory roof are provided to carry off the surplus carbonic acid. These tanks are usually provided with steam heating-coils.

Having limed the juice, carbonic acid is forced into it near the bottom of the tank. The gas attacks the lime forming the carbonate and then forms sucro-carbonates of lime and renders the juice very viscous. It is at this stage that foaming begins and it increases with the increase of sucrocarbonates. There is always danger of forming dark-colored decomposition products with the glucose when steam is used to beat down this foam. Further there may be excessive rise of temperature, which will later be shown to be very objectionable. The carbonic acid gradually combines with the lime. The sound made by the bubbles of gas in the juice and the violence of the frothing are indications to the attendant of the progress of the carbonation.

The temperature gradually rises during the progress of the carbonation. During the early stage the temperature should approximate 45° C. and should nearly reach 55° C. when all the lime is precipitated. When steam is not used to reduce frothing, the rise is not sufficient for the final stage of the process. Therefore, when neutrality of the juice to very sensitive phenolphthalein paper is nearly reached, steam is turned into the heating-coils and the temperature is gradually raised to 55° C. Finally when the phenolphthalein paper (Dupont paper) indicates neutrality the juice is heated to 70° C. It is necessary to raise the temperature in order to break up the sucrocarbonates and to facilitate filtration of the juice. The attendant may note approaching neutrality by the "spoon test," *i.e.*, the appearance of the juice held in a spoon. The precipitates separate sharply from the juice when the latter has an alkalinity equivalent to approximately 0.04 per cent calcium oxide or as often stated 0.4 gram lime per liter. The expression "equivalent" is used here because the alkalinity is partly due to potassium and sodium hydroxides, formed by the action of the lime upon the sodium and potassium salts of the juice. The spoon-test must be followed by frequent tests with the phenolphthalein paper (Dupont paper) until neutrality to this paper is reached. The juice is then heated to nearly 70° C. and is over-carbonated during a very few seconds to neutralize the slight alkalinity arising from lime that has not been attacked by the carbonic acid. In practicing the carbonation process on beet-juices allowance must be made for alkalinity other than due to lime, to avoid over-carbonating. This is not usually necessary in cane work, but it is advisable to make occasional tests for potash alkalinity to be prepared to correct for it.

The next stage of the process is the filter-pressing. The filter-presses are

dress'd with heavy cotton-cloths as in the defecation process. Thin cotton-cloths are usually placed over the heavy cloth to protect it from wear. Three suits of heavy cloth are usually consumed per five suits of the thin. Steam is turned into the press before use and is not shut off until it escapes freely from the juice-cocks. The object of this steaming is to destroy bacteria, which, on account of the low temperature of the material to be filtered, would otherwise be very active in destroying sugar.

The carbonated juice is pumped into the presses at pressures up to about 45 lbs. and should filter very rapidly. The juice should flow very freely from the cocks and the press-cake should be firm and granular. Contrary conditions, sluggish filtration and a pasty press-cake indicate the use of too little lime or an imperfect carbonation. The work of the filter presses is the best indication of correctness of the manipulations. The filter press-cake is usually washed in the press to a low sucrose content. (See under filter presses, page 70.)

The filtered juice is sulphited, concentrated, etc., as in the sulphitation process.

*De Haan's Single Carbonation Process.*—This important modification of the carbonation process is due to J. S. de Haan, Klaten, Java, and is in use in several factories under his technical direction.

This process reduces the consumption of lime and simplifies the equipment without sacrificing the quality of the sugar product. The alkalinity of the juice is kept within very moderate limits during the carbonation.

The raw juice is heated to 45°–50°C., and then a small stream of milk of lime and the carbonic-acid gas are simultaneously turned into it. The flow of the lime and gas is carefully regulated so as to maintain an alkalinity approximating very closely to 0.25 per cent until all the lime required has been added. The usual total quantity of milk of lime of 35.7° Brix is from 4 to 5 per cent of the volume of the juice. Approximately 20 tons of lime-stone and 1.8 tons of gas-coke are used per 1000 tons of cane in producing the lime and carbonic-acid gas. The carbonation is now continued to neutrality to Dupont paper and the juice is heated to 70° C. The injection of the gas is continued one minute after neutrality is reached. The object of this over-carbonation is to prevent particles of lime that have not been converted into the carbonate from rendering the juice alkaline and discoloring it.

The juice is now filter-pressed as in the preceding process. The required filter-cloth area is approximately 1.7 square feet in a frame-press per 1 milling capacity-ton of cane per day.

The filtered juice is concentrated to sirup and, after cooling, is sulphited to an acidity equivalent to 25 to 30 cc. of 100th-normal alkali per 10 cc. of sirup.

**56. Double Carbonation Process.**—All carbonation methods are based upon the original French process used in treating beet-juices. The double process as applied in cane factories differs from the modern beet method only in the temperature of the operation and in carbonating to neutrality to phenolphthalein paper (Dupont paper).

From 7 to 10 per cent by volume of milk of lime of 35.7° Brix is added to the juice, which is warmed to about 113° F. (45° C.). The first carbonation is

then conducted precisely as in the single carbonation method (55) up to the point when the spoon test shows a sharp separation of the precipitate from the juice or a chemical test shows about 0.04 per cent alkalinity. Shortly before this alkalinity is reached the juice is warmed to a temperature of nearly 55° C. and is then filter-pressed. Nothing would be gained by continuing the gassing to a lower alkalinity than 0.04 per cent. With this alkalinity there is no danger of redissolving parts of the precipitates, the juice filters very freely and sufficient lime is left for the second carbonation or saturation.

Lime may or may not be added to the filtered juice from the first carbonation preliminary to the saturation. This juice usually contains sufficient lime except when, through error, the gassing has been pushed too far. The second carbonation proceeds very rapidly, without foaming, and is pushed to neutrality as in the preceding processes. The juice is finally heated to 158° F. (70° C.) preparatory to filtration, and before discharging from the tank should be gassed for a few seconds to prevent deleterious action of particles of caustic lime that may have been occluded in the precipitates.

The saturated juice, on account of the condition of the precipitates, is filtered under very low pressure. Gravity filters or shallow frame-presses are usually used for this purpose with a pressure of but 4 to 5 pounds. (See 63.) The filter press with gravity pressure is the more economical means of filtering.

The object in conducting the process in two stages and filtering the strongly alkaline juice from the first is the removal of substances that are soluble or slightly so in neutral solution. Among these substances are magnesia, usually largely derived from the limestone, the oxalates and possibly other organic salts and substances, such as coloring matters, that are held mechanically by the precipitates.

The precipitates from the second filtration are usually mixed with the juice going to the first filter-presses and are thus subjected to washing with the press-cake.

Sulphitation of the filtered juice from the second carbonation is practiced as in the previous processes and as recommended by Harloff in the following paragraph, and it is then concentrated to sirup. The sirup, after cooling, is usually sulphited to an acidity equivalent to from 15 to 30 cc. of N/100 alkali per 10 cc. of sirup.

**57. Harloff's Acid Thin-juice Process.**—Harloff's experience in Java led him to a study of the influence upon the juice, sirup, and consequently the sugar of various salts, especially the potassium sulphites and carbonates and the corresponding salts of lime and iron. These studies led to a very general practice of sulphiting the raw juice or the carbonated juice to acidity to phenolphthalein in the Java factories. Dr. Spencer visited many factories in Java in the summer of 1913, and, with very few exceptions all sulphited to acidity. It had long been the custom to sulphite sirup to acidity, but not the juice.

Of most importance, Harloff <sup>20</sup> found that sulphites do not darken clarified juice containing glucose on heating and that carbonates do. This led to the natural conclusion that in so far as may be all the salts should be converted into

<sup>20</sup> "Plantation White Sugar Manufacture." W. H. Th. Harloff.

sulphites, since it is impracticable to remove them. He accomplishes this by sulphiting to neutrality to litmus, which corresponds to slight acidity to phenolphthalein. Further, iron salts, if present in the raw or carbonated juice, are in the ferric state. These salts are reduced to the ferrous state in juices by the sulphurous acid and are colorless and remain so in acid solution. The ferrous salts do not crystallize out with the sugar from acid solution.

Litmus cannot be conveniently used at night or when the light is poor, and therefore, is an uncertain instrument in the hands of a laborer. Harloff recommends the use of the Vivien tube, which is employed largely in France in beet-sugar work. The tube is filled to the zero mark with N/100 potassium hydroxide containing phenolphthalein, and sulphited juice is added until the red color is discharged. If, for example, the requisite quantity of juice to discharge the color is 10 of the scale on the Vivien tube and a smaller number is obtained, the juice is too acid and the injection of sulphurous acid must be reduced. Check tests should occasionally be made with juice neutralized to litmus in daylight. Obviously a burette control could be used, but this is not quite so easy a manipulation as with the Vivien tube. pH control is now being used as in other clarification processes.

Harloff recommends that the juice mixed with the precipitates be not heated to a higher temperature than 90° C. (194° F.) in closed heaters on account of fouling the heating-surfaces. Heating above this temperature should be in open tanks.

It has been the custom for a great many years in Louisiana to work with an acid juice simply because with this condition the color of the sugar is better, and apparently with no definite idea as to why this is true. Harloff's investigations show very clearly why the sugar is better.

Harloff calls attention to the corrosion of the tubes of the evaporators, on the vapor side, when the juice is left acid to litmus. He also states that the return waters are acid under this condition and damage the tubes of the steam boilers. The boiler-feed water should be rendered slightly alkaline with soda when sulphitation is practiced.

**58. Plantation White Sugar with Vegetable Carbons.**—The literature on this subject is voluminous and is largely made up of claims and descriptions of processes by carbon manufacturers and inventors. With no intention of belittling its importance, the scope of this book does not permit of a detailed treatment of the subject since no one of the carbons has gained especial favor in this type of work, nor is any appreciable amount of white sugar made in this way. In general, the juice is first defecated, the reaction being left slightly acid (pH 6.4-6.8) and the carbon added to the juice which is then filter-pressed. Other processes add the carbon to the sirup (meladura) as it leaves the evaporator. The decolorization and gum-removal are high with all the better vegetable carbons, the main difficulty being the revivification and the cost of the carbon itself.

The refining of melted raw sugars by the "Suchar Process" (p. 175) is an established process which is described in the chapter on Refining.

## SPECIAL APPARATUS USED IN THE SULPHITATION AND CARBONATION PROCESSES

**59. Sulphur Stoves or Ovens and Sulphiters.**—The older type of stove, Fig. 25, is used quite generally in Louisiana. The body of the stove is water-jacketed for cooling. A large pipe, usually 6 to 8 inches in diameter, also water-jacketed, leads about 4 to 6 feet to the gas-main. This pipe serves as a subliming-chamber and catches a large part of the sublimed sulphur. Suitable valves are provided for disconnecting the stove from the main pipe-line so that a stove may be cleaned without interfering with the others in the battery. Air for the combustion of the sulphur should be drawn through a drying-box and then forced into the stove by a compressor.

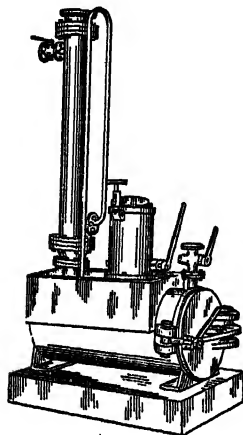


FIG. 25.—Sulphur Burner.

It is very essential that this sulphur-stove be provided perfectly dry, clean air for the combustion in order to avoid partial oxidation to sulphuric acid.

The drier is preferably a cast-iron box provided with a tight-fitting door and trays for holding layers of quick-lime. The air enters the box at the bottom and passes over the lime on its way to the compressor. A drier about  $3 \times 3 \times 4$  feet is usually large enough for a stove of 8 square feet fire area. Two stoves of this size are sufficient for a factory consuming about 750 lbs. of sulphur per day.

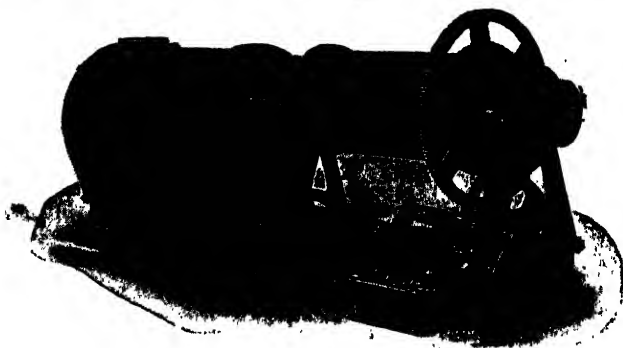


FIG. 26.—Rotary Sulphur Burner.

A rotary sulphur burner, made by the Glens Falls Machine Works (Fig. 26) is in use in beet factories and the more modern cane installations which use sulphur. This burner uses induced draft. The sulphur is fed to the rotary burner by a mechanical feed (not shown in the cut) and melts by its own heat of combustion in the rotating cylinder presenting a large surface for combustion as the sulphur drips through the air. Air is drawn in at an adjustable neck ring and "Anti-sublimation Sleeve" at the connection between the rotating



drum and the combustion chamber shown at the left. This is a cast-iron or brick-lined compartment with baffles in which the oxidation of the sulphur and mixing with the diluting air are completed. A uniform gas (5 per cent to 16 percent  $\text{SO}_2$  as desired) free of sulphuric acid is delivered to the sulphitators.

Sulphitation tanks are of many forms, but the usual one is a moderately deep iron-tank provided with a cover and chimney to the outer air, suitable test-cocks, valves, and a perforated pipe for distributing the sulphurous-acid gas. The pipes and tanks should be arranged to facilitate cleaning at frequent intervals. The bottom is preferably conical. Perforated pipes for steam-jets to break down foam should be provided in sirup sulphitators. Where the tank is used to saturate large quantities of lime, as in Bach's process, intermittent work is advisable, and at least three tanks should be installed, otherwise a single continuous sulphitation should be used. Two tanks may be used in this method, though one very deep one with more careful manipulation will answer. The juice enters the first tank at the bottom and is sulphited to approximately the desired test. It overflows and enters the second tank at the bottom and is sulphited to the required acidity. From the second tank the juice flows through juice-heaters and filters to the charge-tanks of the evaporator.

**60. Carbonation-tanks.**—The carbonation-tanks are of iron and should be more than 20 feet in depth for the first carbonation to take care of foaming. The tank should have sufficient steam-coil capacity to heat the juice quickly. The carbonic-acid and juice-connections should be large to provide for rapid work. A first carbonation should require about ten minutes and a second about three to five minutes for the gassing.

Formerly the gas was led into the juice through perforated pipes. Such pipes always give trouble through incrustations of lime. In more recent practice the gas enters through a large pipe in the conical bottom of the tank and is deflected at intervals by baffle-plates or other device, so arranged as to insure thorough distribution of the carbonic acid. There are many methods of arranging the pipes to reduce the scaling and facilitate cleaning them.

Continuous carbonation is advisable for the second stage of the process. The quantity of lime to be saturated is very small and with a suitable device the outflowing juice is readily carbonated to the desired point. There are several continuous carbonators on the market designed for beet work. Descriptions of them will be found in many works on beet-sugar.

If intermittent second carbonation is practiced, the tanks may be comparatively shallow, as there is little foaming.

**61. Lime-kilns.**—The carbonic acid is obtained from lime-stone calcined by coke-fires in a special continuous kiln. This kiln, as also all other carbonation and sulphitation machinery are the identical devices that are used in beet-sugar manufacture.

The kiln proper is 25 to 30 feet high and is the frustum of a cone of narrow angle with the small end upward. A mixture of limestone and coke is fed into the kiln at the top through a conical self-closing door. Three distinct zones are maintained in the kiln, viz.: (1) at the top, the fresh zone and unignited coke; (2) at the middle is the combustion and dissociation zone; (3) below the combustion-zone is that in which the resultant lime is cooled and drawn off.

Kilns are usually built of the Belgian type in which the body is supported

upon four short columns, leaving the bottom open and free for the discharge of lime. The columns stand upon a concrete platform upon which the lime rests. Stone is piled upon this platform to support the kindling and mixture of stone and coke, when the kiln is put into commission. As the firing progresses and the lime is produced this stone is removed from time to time and finally its place is taken by quick-lime. The kiln is provided with numerous openings, having tight-fitting plates or doors, for use in watching the progress of the firing and for breaking down "scaffolds."

The draft is induced by the carbonic acid pump, which is located conveniently near the carbonation tanks. A pipe leads the gas through a washer and scrubber and thence to the pump which discharges it into the carbonation-tanks. The gas is thoroughly washed with water.

The kiln must have but one inlet for air, and that where the lime is discharged. The quantity of air drawn in, and, therefore, the combustion, is regulated by the speed of the carbonic-acid pump. It is evident that this pump must be kept in thorough order and must work with great regularity, since the success of the carbonation depends upon it. The zone of dissociation must be maintained in its proper place. The essential conditions are the delivery of rich gas containing above 30 per cent of carbonic acid and properly burned lime.

The gas should be frequently tested as described in 417, 418. The quality of the coke and stone<sup>30</sup> must be controlled. The following brief summary of kiln conditions will assist in the interpretation of analyses:

(1) The gas contains a large excess of oxygen, little carbonic oxide, and a low percentage of carbonic acid: Leakage is indicated at a point between the gas-pump and the kiln. If the combustion zone is white hot the gas-pump is running too fast or the coke is too coarse. The draw of lime should be increased.

(2) The gas contains too little carbonic acid and neither carbonic oxide nor oxygen is excessive: Smaller quantities of lime should be drawn and at longer intervals. The mixture of stone and coke should be investigated, as the proportion of the latter may be too large.

(3) The richness of the gas in carbonic acid is fluctuating: The pump may be running too slowly and irregularly.

(4) The gas contains an excess of carbonic oxide and normal quantity of oxygen: The combustion of the coke is incomplete and carbonic acid is converted into monoxide on account of deficiency of oxygen; other conditions being normal, the coke is too coarse and the pump is running too fast.

(5) The gas contains both oxygen and carbonic oxide in excess: The circulation of the gas is slow and the sampler is drawing some air.

The American beet-sugar factories usually use high-grade metallurgical coke in their kilns. The high cost of such coke has forced the Java factories to substitute the cheaper gas-coke and oil-still residuum. The residuum is used in the ratio of 1 to 12 of stone by the weight. Coke is used in ratio of 1 to 9.5-11 of stone.

<sup>30</sup> See analysis of lime-stone, p. 391.

## CHAPTER VII

### FILTRATION PROCESSES AND MACHINERY

**62. Juice Filtration.**—In the usual processes of cane-sugar manufacture the entire clarified juice is not filter-pressed, but only that portion contained in the scums and precipitates from the defecation. The juice clarified by the defecation process contains fine flocculent matter which soon obstructs the pores of the cloth. Several processes, depending upon the addition of sawdust, lignite, charcoal, kieselguhr, etc., to the clarified juice preparatory to filtration, have been devised, but none has been economically successful. In the Bach sulphitation process, both the juice and sirup may be filter-pressed. The precipitated calcium sulphite supplies a filtering medium as does the carbonate in the carbonation processes.

Mechanical filters of many types, using sand, cloth, or other filtering media have been used with varying success. Bone-black filters were formerly used in making plantation white sugars, but these have been displaced by the sulphurous-acid processes.

In practically all raw sugar manufacture, the clarified juice decanted from the defecators is sent direct to the multiple effect evaporators without further treatment. If the clarification has been defective no amount of screening will greatly improve the juice nor will any form of filtration give satisfactory results. A mechanical screening through fine wire cloth is effective in removing floating particles of bagacillo and other suspended matter which the defecating precipitate has failed to enmesh, but even this is not generally resorted to. A  $100 \times 100$  metal cloth is employed frequently but there are difficulties in keeping the screen from stopping up. Geerligns<sup>1</sup> describes a slanting bronze gauze screen containing 2000 meshes per square centimeter (about 13,000 per square inch) as well as a rotating screen similar to the Peck strainer (Sec. 29). The Mitchell electric vibrating screen has just come into use for this purpose at some Cuban factories.

A device patented by Hadfield in Hawaii<sup>2</sup> consists of a screened compartment inside a square shallow tank, the screening surface being  $150 \times 150$  monel metal cloth. The clarified juice flows into the tank outside the screened compartment and then finds its way through the fine screen. This would very quickly block up were it not for a spray of strained juice which is gently played on the outflow side of the screen. The idea is not to dislodge the particles of bagacillo, sediment, dirt, etc., which are caught on the screen, as this is taken

<sup>1</sup> "Cane Sugar Manufacture," 2nd Edition, London, 1924.

<sup>2</sup> W. G. Hall. *The Planter*, 72 (1926), 290.

advantage of as a filtering medium. The spray is the principal part of the system. The theory is explained as follows:

"If a perfectly flat piece of very fine-meshed screen is held horizontally, and a drop or so of water gently poured also horizontally over the surface it will be seen that the water remains on the surface and does not penetrate the meshes of the screen. A slight canting of the screen will cause the drop of water to roll off the surface like water off a duck's back. If, however, the screen is touched with the finger or sprayed with water, directly under this drop, it at once penetrates the meshes of the screen. Some such attraction, whether capillary or otherwise, is going on whilst the screens are working, for, once the spray stops the impurities lodging on the outside of the screen block the system. Start the spray again, and the whole system again functions."

A factory running 50 tons of raw juice per hour is said to require three tanks containing 24 square feet of screen surface each. Cleaning and washing takes but a few minutes, the frequency depending on the cleanliness of the juice.

**63. Mechanical Cloth Filters.**—The use of bag filters such as are used in refining (Sec. 162) is rarely resorted to in the defecation processes. Occasionally in white sugar work use is made of this form of filter (Sec. 56) but only when some other reagent is used in addition to lime.

Mechanical cloth filters of the Danek type are also rarely used. They consist of a large number of rectangular bags suspended in a closed iron box. Each bag or pocket has an independent discharge-pipe communicating with the inside of it. A metal distender prevents the bags from collapsing. The clarified juice flows into the filter by gravity, under a low head, fills the box, and filters into the bags. The mud collects on the outside of the bags and from time to time falls off. The pressure is so low that the mud is not impacted upon the cloth. When the cloths become foul, the flow of juice is shut off and the mud is sent to the filter-presses.

**64. Sand Filters.**—Several types of sand filters for juice and sirup are in use. As the name of the filter implies, the filtering medium is fine, sharp sand. Pulverized coke may also be used. The filtering material must be of grains of uniform size, otherwise the filter will soon clog.

When the filtering medium is clogged with mud the juice it contains is displaced with water and the sand is then thoroughly washed with hot water under pump-pressure or otherwise, according to the type of the filter. After washing the filter is again ready for service.

These filters have been used with moderate success in the cane-industry, but their use is not general. Geerligs<sup>3</sup> reports the removal of considerable cane fiber, albuminoids, wax and mineral matter by sand filters.

**65. Excelsior Filters, Bagasse Filters, etc.**—*Excelsior Filters.*—The excelsior filter was devised in the Hawaiian Islands for filtering clarified juice. A small tank, about 2.5 to 3 feet deep and of convenient dimensions for manipulating the filtering medium, is fitted with a false bottom about 2 inches above its bottom. A pipe-line for bringing clarified juice to the filter is connected with it at the center of the tank's bottom and over this inlet is

<sup>3</sup> "Cane Sugar Manufacture." 2nd Edition, 1924, London.

placed a small baffle-plate to break the force of the current and distribute the juice. An overflow-pipe is connected near the upper edge of the tank to lead the filtered juice to the charge-tanks of the evaporators.

The filter is prepared for work by packing it with the ordinary excelsior that is used in shipping merchandise. A wire screen is placed on top of the excelsior to prevent it from floating and to retain particles of the material that might be carried along with the juice. A filter capacity of approximately 150 cubic feet of excelsior is necessary per 1000 tons of cane. These filters and others of a similar type have been largely discontinued because of the great heat losses inherent in their use and also because of the possibilities of fermentation and inversion. It is now recognized that the more rapidly the juice can be brought to the sirup stage the better.

*Bagasse Filters.*—These filters are constructed in precisely the same way as the excelsior filters except that fine bagasse is the filtering medium. When the filtration becomes sluggish the bagasse is removed from the filter and put upon the mill-carrier for regrinding.

This material has a great tendency to ferment and thus contaminate the juices during filtration.

*Fiber Filters, etc.*—Various fibers are used to a limited extent in a way similar to excelsior. Thin sheets of paper-pulp have found a small application in sirup-filters. Granulated cork, asbestos and many other materials have been used but with little success.

*Alcohol Precipitation.*—In a very old French process, alcohol was used as a precipitant in sorghum-juice. Filtration followed without difficulty. The U. S. Department of Agriculture modified this process in adding an equal volume of strong alcohol to sorghum-sirup of about 55° Brix. A very heavy precipitate consisting partly of gums was thrown down and was very easily removed by filter-pressing. The alcohol was recovered by distillation and the sugar was crystallized as is customary.

*Centrifugal Separators.*—Several forms of centrifugal separators have been brought out by inventors from time to time. In these the machine differs from the ordinary centrifugal in having no perforations in the basket. The defecated juice or even juice without previous removal of the mud (cachaza) is run into the machine near the bottom of the basket. The mud is thrown to the wall of the basket by the centrifugal force and the clean juice flows over the rim.

The cost of the plant and the expense for power, loss of heat, oil, etc., are possibly the reasons for the small extension of this process. If the defecation is good and the separation of the precipitates therefore sharp, the centrifugals will deliver very clean clarified juice.

**66. Filtration of Scums and Muds.**—The scums and juice tank-bottoms may be easily filter-pressed. It is advisable to heat this material thoroughly in steam "blowups," settle the mud, and decant the clean juice preparatory to filtration. There is a gain both in sugar and facility of filtration by this treatment. There is an additional gain of sugar by adding water to the residual mud and again heating and settling it, and decanting the thin juice. The mud filter-presses more readily when alkaline, therefore lime is usually added to it to

bring it to a pH of 8.0-8.5. However, great care should be taken to avoid an excess of lime as is explained later.

**67. Filter-presses.**—Filter-presses are so generally used in the sugar industry that full description of them is unnecessary. Briefly, a filter-press consists of a number of iron plates and frames or recessed plates over which filtering cloths are placed. The frames and plates are supported on a heavy framework and are clamped together by a powerful jack-screw. The cloth itself makes the joints between the frames and plates.

There are two general types of presses, the center-feed and side-feed or frame presses.

The center-feed presses are made up of heavy recessed plates, with a round opening in each and a corresponding hole in the cloth to form the inlet channel. The cloth is clamped to the plate at each of these openings.

There is no hole in the cloth in the frame-press. A lug projects from each frame and plate and in each there is an opening to form the mud-channel. Rubber rings or cloth "stockings" form the joints between the lugs.

When a press is in use the mud collects in the frames or recessed plates and the filtered juice runs off through suitable openings.

A pressure of 40 pounds to the square inch is sufficient for very good press-work. With heavier pressure there is risk of breaking the presses and liability of forming a coating of almost impervious material upon the cloths.

It is the usual practice in making white-sugar to filter-press the mud without liming it. It is often advisable to use a moderate quantity of lime, and if necessary reclarify the filtered juice, mixing it with clarified juice, or acidulating it with phosphoric acid.

**68. Washing the Cake.**—Filter-presses are usually arranged so that the press-cake may be washed or the juice it contains be displaced with water. In displacing the juice the cocks are closed on the water-plates and water is forced through the cake, driving the juice ahead of it. This is feasible only in defecation factories having very large filter-press capacity, as the displacement is exceedingly slow. This method is applicable in the carbonation and certain of the sulphitation processes. In order to economize wash-water and consequently fuel for evaporating, the last runnings of a previous press are often used in beginning the washing of the cake.

Galbis and Bach devised a scheme for washing presses which is simple, rapid and practical. The supply of scum juice is shut off from the press before the frames have completely filled with press cake so that an open space is left between the two inner surfaces. The current of wash-water is run through the same channels as the juice and this open space permits free access of the water to all parts of the cloths. This scheme is only applicable where a firm, hard cake is found. Hot water is used in sweetening off at the same pressure as the juice when the filtration was stopped.

Geerligs<sup>4</sup> says that in practical working of this scheme the sweetening off is stopped when the effluent reaches a density of 2 to 3° Brix, the remaining liquid in the cake being expelled by compressed air or steam. He reports an average filtration time of one and one-half to two hours and one-half hour for

<sup>4</sup> "Cane Sugar Manufacture." 2nd Edition, page 180, 1924.

sweetening off and steaming, the sucrose content of the cake being reduced to under 1 per cent. Experience in Cuba has shown that washed cake is extremely variable in composition when sampled in different parts of the frames. General practice on the island is to dilute muds and not to wash.

**69. Double Filter-Pressing.**—Double filter-pressing is a method of exhausting the press-cake of sugar in the defecation and other processes but with certain marked disadvantages as mentioned below. The first pressing is conducted as usual. The first press-cake is conveyed to a machine for breaking it and then it is beaten to a cream with water. This cream is next heated to boiling and is then pumped into a second set of filter-presses. The filtrate from these presses may be used in diluting mud (cachaza) preparatory to the first pressing. This method requires a filter-press plant of sufficient capacity to devote about one-third of the presses to the second filtration. The sucrose content of the press-cake may readily be reduced below one-half per cent without excessive use of water.

Many factories which adopted double pressing have discontinued it because of the conviction that the gain in sucrose was not sufficient to offset the redissolved impurities put back into the process. Allen <sup>5</sup> published results to show that no such adverse conditions could be ascribed to double pressing, but Keane, McCalip and Paine <sup>6</sup> in a similar study concluded "that it seems debatable whether the sugar recovered is of sufficient value to offset the effect of the harmful impurities which enter the process in this operation." The factories which discontinued double pressing have done so largely on their own experience in this matter of washing out excess of impurities for return to the process, rather than on the findings of others.

**70. Leaf Filters.**—Kelly, Sweetland and Vallez Filters similar in general form to those used in refining (Sec. 163) have found some use in the raw sugar industry though they have not succeeded in displacing the simpler plate-and-frame presses to any considerable extent.

**71. Quantity of Press Cake.**—The proportion of filter press cake varies greatly with the locality, efficiency of milling, and juice straining and press efficiency. The quantity of lime used in the clarification has also an effect on the proportion, the more lime, the more press cake. The addition of earth from flooded canes also modifies the proportion.

The amount of cake per ton of cane gradually increases as the season advances and is at its maximum at the end of the grinding. Washing the cake reduces the weight not only through the removal of sugar but also by the washing out of soluble impurities. Double filter pressing produces less cake. Too short a press cycle, resulting in cakes with high moisture percentage is a frequent cause of excessive quantity and attendant loss of sugar. An example of this is a factory that usually produced a crop average of 35 to 40 pounds per ton of cane which reduced this number to about 31 pounds by lengthening the time under pressure. The cake per ton of cane ranges from 15 to 40 pounds in Cuba, with efficient factory work. Allen <sup>7</sup> gives the following figures for single

<sup>5</sup> The Planter. 71 (1923), 214.

<sup>6</sup> "Facts About Sugar." October 8, 1927.

<sup>7</sup> The Planter. Vol. 71 (1923), page 250.

and double pressing covering six years of each class of work in a Porto Rican factory:

	Per Cent Sugar	Per Cent Moisture	Pounds Cake per Ton of Cane	Square Feet Filtering Area per 1000 Tons Cane
Single pressing . . . . .	3.925	55.15	25.25	2206
Double pressing . . . . .	1.233	59.03	27.40	3156

*Note:*—The greater weight of cake with double pressing is accounted for in this instance by the higher moisture content. Similar figures for a large Cuban factory showed:

	Per Cent Sugar	Pounds Cake per Ton of Cane	Purity of Press Juice
Single pressing . . . . .	5.08	33.60	81.2
Double pressing . . . . .	1.90	28.00	72.5

**72. Disposal of Filter Press Juice.**—The filtered scum juice from the presses may be disposed of in one of several ways: (1) by clarifying and resettleing separately and then mixing with the clarified juice; (2) by returning to the defecators for redefecation and resettleing in conjunction with the main stream of raw juice and (3) by pumping it direct to the charge tanks of the evaporators.

The first method (separate clarification) is rarely resorted to and the third method of mixing the material with the defecated juice is objectionable if excess lime has been added to the scums before filter pressing. Under these circumstances the more alkaline press juice may set up a slight precipitate in the defecated juice and this precipitate, in combination with the excess soluble lime increases the scaling of evaporator tubes. The second method of sending the press water back through the clarification system is general practice and until recently was considered to offer no objections whatever since it was believed that any excess lime would react with the undefecated juice and be an aid to clarification.

In the work already cited, Keane, McCalip and Paine<sup>8</sup> have shown that this action does not take place and that 80 per cent of the excess lime in the filtered press juice finds its way into the evaporator even though it is mixed

<sup>8</sup> Ind. Eng. Chem. Vol. 20 (1928), page 373.



with the raw juice and redefecated and that all the reversible colloids formed by the action of lime (see Sec. 42) pass through the redefecation untouched and go to the evaporators exactly the same as if the press juice had been sent direct to the evaporator instead of passing the second time through the defecation system.

This work indicates the necessity of a careful control of the liming of the scum waters and the avoidance of the addition of any lime at this station if a good cake can be secured without it. In Hawaii, with the more alkaline clarification recently adopted, it has been found that no lime is needed at the press station, and the press water is sent directly to the evaporators according to the third method given above. It is probable that returning the press juice to the undefecated juice for retreatment (Method 2) is to be preferred even with correct lime control in order to avoid the possibility of muddy press juice (caused by some failure at the presses) being added to the clarified juice going to the multiple effects.

## CHAPTER VIII

### CHEMICAL REAGENTS USED IN PURIFYING THE JUICE<sup>1</sup>

**73. Lime.**—The most effective agent of moderate cost that has been found for the treatment of the juice is lime. There are several methods of preparing this substance for use in defecation, the commonest and most convenient practice being to prepare from quick-lime a milk of lime of 12° to 15° Baumé (approximately 10 to 13 per cent CaO) which is pumped to the liming station through pipe-lines in which it is kept constantly circulating. Other methods less commonly used follow: (1) Quicklime is ground to a powder which must be slaked with water before liming the juice. (2) Dry slaked lime is prepared by sprinkling water on heaps of lime and the sifted powder mixed with *juice* preparatory to use in the defecation. (3) The lime is slaked in large tanks and then allowed to settle to form a heavy paste, after which the supernatant water is drawn off, and the paste weighed for use in the liming tanks.

The preparation of the lime is of great importance, especially in the manufacture of white sugar. Essential conditions in properly slaking lime are that full time be allowed for the reaction and that too much water be not added, thus reducing the temperature. The slaking tanks should be large so as to retain the heat of the reaction and promote the slaking.

When the factories of Louisiana were very small, it was the custom to slake the lime in advance of the grinding season. The milk was reduced, after slaking, to a low density with water and then passed through fine screens into a second tank and so on until all the containers were filled. After allowing a few days for the hydrated lime to settle out, the supernatant liquid was drawn off and fresh portions of the strained milk were added to the paste already in the tank. The alternate drainage and settling were repeated until all the tanks were filled with the heavy lime-paste. Dr. Spencer frequently noted indications of further slaking even two or three weeks after the tanks had been filled. This description is given to emphasize the importance of the time element.

**Quality of Lime.**—The lime used in the purification of the juice should be very pure. The magnesia and soluble silicates of the lime used in the defecation processes form scale in the evaporators. However, mention is made farther on of the satisfactory use of lime containing large quantities of magnesia.

Geerligs<sup>2</sup> gives the following tests for a good lime for clarification purposes: It should become very hot in a few minutes when treated with half its weight

<sup>1</sup> A list of several hundred substances and combinations that have been proposed for use in sugar manufacture, is given on page 535.

<sup>2</sup> "Cane Sugar Manufacture." 2nd Edition, London, 1924.

of water; should form a soft cream after slaking when mixed with ten times its weight of water; this cream should not contain more than one-tenth of the original weight of lumps which fail to pass through a fine sieve, and most of these particles should soften in an hour's time. He places the maximum allowable percentage of silica, of oxides of iron and alumina and of magnesia at 2 per cent each.

Hydrated lime is sold in powdered form packed in heavy paper sacks such as are used for cement. The convenience, cleanliness and purity of lime in this form have led to its adoption in most refineries and in cane factories in Louisiana and other sections where the freight rates will permit. Its use in the British West Indies is on the increase. A hydrated lime from the southern part of the United States (calculated to non-volatile basis) showed:

Total CaO.....	96.80
MgO.....	1.20
SO <sub>3</sub> .....	.09
Iron and alumina.....	1.11
Insoluble and undetermined.....	.80
	<hr/>
	100.00

The quality of the limestone used in the carbonation process is of great importance. Certain stones rich in silica tend to "scaffold" in the kiln, and others have hydraulic properties, resulting in an almost impervious filter-cake.

Lime from coral beach-sand is used to some extent in the Hawaiian factories. The following analysis<sup>3</sup> of lime from this source shows high magnesia content: Silica, 0.18 per cent; lime, 91.7 per cent; magnesia, 4.15 per cent. Analyses of two Cuban coral sands (dry) gave a magnesia carbonate content of 6.6 and 4.5 per cent. The first of these Cuban sands would produce lime containing 92.5 per cent calcium oxide, 6.0 per cent magnesium oxide and 1.5 per cent silica, etc.

The following information relative to coral sand is from the Paia factory, Island of Maui, H. T.<sup>4</sup>: "Coral sand is burned in rotary kilns with temperature control. Damp sand is preferred, since the temperature at which dissociation of carbon dioxide takes place is slightly lessened in the presence of steam generated from the moisture.

"Coral sand contains considerable magnesia which is usually presumed to be objectionable in the defecation owing to its depositing upon the heating-surfaces. Further, a portion of the magnesia remaining in the molasses would increase the molasses output with consequent loss of sucrose. The experience of the Maui Agricultural Co., Paia factory, showed a large elimination of the magnesia in the press-cake. The defecation was good, the yield of sugar satisfactory and the output of molasses was small. The lime contained 5.45 per cent of magnesia." Dr. Spencer visited Paia in 1913 and saw coral lime in use in the defecation process. The alkaline filtration in the double carbonation process eliminates the magnesia from the juice.

<sup>3</sup> Spec. Report, July, 1913, Expt. Sta. Haw. Sugar Planters' Ass'n.

<sup>4</sup> Planters' Monthly (Hawaiian), Nov., 1909, 444.

**74. Sulphurous Acid.**—The production is described in the white-sugar section, page 64. This reagent is little used in raw sugar manufacture except in Louisiana.

Aside from its bleaching effects the sulphurous acid produces a heavy precipitate with the lime which assists mechanically in the clarification. It also breaks up some of the lime salts and thus reduces the viscosity of the sirup and massecuites. Sulphured sirups and massecuites boil much more freely in the vacuum-pan than those made without this reagent.

**75. Carbonate of Soda ("Soda Ash"), Caustic Soda.**—Juices which have begun to ferment are better neutralized with soda, rather than with lime, as the latter produces soluble salts that are very objectionable. The use of soda ash to neutralize excess acidity in juice from burned cane is common practice in many factories. The soda salts are also useful in neutralizing molasses. The carbonate apparently has a beneficial effect when taken into the pan in boiling low-grade massecuites.

Carbonate of soda is an incomplete precipitant of lime from its salts in sugar solutions. In a series of experiments de Grobert<sup>5</sup> found that the addition of carbonate of soda to a sirup in the proportion of its equivalent of lime precipitated about 52 per cent of the lime. The addition of two equivalents more of soda precipitated 81 per cent of the lime. A part of the soda remained in a free state and the remainder was neutralized by the organic non-sugar. M. de Grobert states that if carbonate of soda is used in juices, sirups, etc., it should be the theoretical quantity required to combine with the lime.

L. R. Cook<sup>6</sup> cites an experience in pan-boiling in beet-sugar work when an excessive amount of lime salts in the low-grade material prevented the pan from boiling. Cottonseed oil was injected into the massecuite and improved the conditions. The oil was then saponified with caustic soda. This immediately further improved the conditions and the pan boiled freely.

A viscous or very heavy massecuite that is difficult to pump usually yields readily after the addition of carbonate of soda. Such massecuites are more easily purged in the centrifugals after the soda treatment.

S. S. Peck<sup>7</sup> advises the addition of sodium carbonate to a raw juice that contains excessive quantities of sulphate which tends to form calcium sulphate scales in the heaters and evaporators. The sodium carbonate decreases the amount of insoluble ash, phosphoric acid and lime in the filtered juice; it partially removes magnesia; it increases the amount of material removed by the filter-presses and effects a slight increase in the proportion of organic impurities removed from the juice. The use of carbonate of soda materially increases the cost of the defecation.

**76. Phosphoric Acid.**—Phosphoric acid and calcium acid phosphate, under various trade names, are used to a large extent in Louisiana in the clarification of the juice, as a sugar-wash at the centrifugals, and in the preparation of molasses for reboiling.

In the clarification, the phosphoric acid or the phosphate is added in small

<sup>5</sup> Eighth Cong. App. Chem., 8, 21.

<sup>6</sup> *Op. Cit.*, 27, 61.

<sup>7</sup> Bul. 33, Expt. Sta. Haw. Planters' Ass'n.

excess to the slightly over-limed clarified juice. The juice is then boiled and brushed, and run into the settling-tanks. This is the basis of Horne's "super-defecation" process (50).

**77. Various Chemicals Used in the Defecation of the Juice.**—A large number of substances have been proposed for use in purifying the juice but only a few have to a very limited extent been practically applied in manufacture. Among these substances are barytes, hyposulphurous acid (made by the reduction of sulphurous acid with zinc or tin dust), hydrosulphites of sodium and of calcium, clay, alumina, etc.

Hyposulphurous acid is used in bleaching molasses, also tin salt, or muriate of tin. The last named was used in centrifugal washes for yellow sugars, but is objectionable because of its toxic properties.

Kieselguhr (diatomaceous earth, best known under the trade name of Filter-Cel) is used as a filtering medium. The addition of this material to juice or sirup greatly facilitates filtration although its use has become general only in the refining branch of the industry (see page 154).

## CHAPTER IX

### EVAPORATION OF THE JUICE<sup>1</sup>

**78. Multiple-effect Evaporation.**—In the old processes of sugar-manufacture, the juice was evaporated to a sirup and the sirup to the point of crystallization, in open kettles over a fire or in open pans in which the liquor was boiled by live steam-coils. These processes have nearly disappeared and are used only in very small factories in places where the price of sugar or molasses is very high, or where local conditions permit.

In all modern factories the clarified juice is evaporated to a sirup of 55° or 60° Brix in multiple-effect evaporators, the steam being made to do duty two or more times according to the type of evaporator. The evaporation is conducted in various forms of multiple-effect vacuum apparatus of which the so-called "standard" type, in triple, quadruple, or even quintuple effect, is the most generally used.

**79. The "Standard" Type of Multiple Evaporator.**—A description of this form of evaporator in triple effect will serve the purposes of this book, though quadruples are now much more common than triples, and, as will be explained later, vapor cells and pre-evaporators are used in addition in many modern factories. In the "standard" effect to be described the liquor is boiled in a deep layer, from 3 to 4 feet in depth, whereas in shallow and film evaporators the depth varies from a few inches to but a thin film, according to the type of the evaporator.

The standard triple-effect consists of three vertical cylindrical vessels, termed "pans," "effects" or "bodies," the lower part of each of which is provided with a steam-drum fitted with copper or brass tubes, through which the juice circulates and which are surrounded by steam. The steam-drum is termed the "calandria." In the center of the calandria is a large tube which carries the juice back to the lower part of the pans as it boils up through the tubes. This large tube is called the "down-take."

The space above the calandria, the vapor-space, is connected by means of a large vapor-pipe with the steam-space of the second pan, and the vapor-space of the second pan with the calandria of the third. The vapor-space of the third pan is connected by a large pipe with the condenser and vacuum-pump. The juice in the first pan is boiled by the exhaust steam from the various pumps and engines and its water of condensation flows through a trap to the boiler-feed water-tank.

The vapor generated in the first pan is used to boil the juice in the second, and that generated in the second to boil the sirup in the third pan. The juice

<sup>1</sup> See Chap. XII, "Evaporating and Juice Heating," by Prof. W. H. P. Creighton.

is fed into the first pan and circulates from pan to pan and finally reaches the third, the thin juice being admitted as fast as the evaporation of the water and the removal of finished sirup permits. A vacuum is produced in the third pan by the pump and condenser and in the first and second pans by the condensation of the vapors in the calandrias of the second and third pans. The vacuum in each pan depends somewhat upon the working conditions, but is usually about 5 to 7 inches of mercury in the first, 14 to 17 in the second, and 26 to 28 in the third pan. By reason of the differences in the vacuum, the juice travels from pan to pan and the vapors produced in the evaporation in the first pan are hot enough to boil the liquor in the second, and those in the second to boil the sirup in the third pan. The condensation water of the second pan is either drawn into the calandria of the third pan, or is directly pumped to the boiler-feed tanks, and that of the third pan is also removed by a pump.

These pumps are usually termed "sweet-water pumps." The sirup is removed from the third pan continuously by means of a pump, the workman regulating its density by the steam-pressure and the quantity of juice in the first pan, and the rate of pumping the liquor from the third pan. Valves are provided on the pipes connecting the pans to regulate the flow of the liquor.

The procedure described is sometimes somewhat modified by the use of Chapman's circulating-pipes. These pipes are like inverted syphons connecting the liquor-spaces and are of such length that the vacuum in the pans is not sufficient to empty them. They are arranged to maintain a constant level of liquor in the pans without the use of other regulating-valves than those for the juice and steam in the first pan. These pipes cause the liquor to circulate very rapidly through the tubes of the calandria. In operating the apparatus the workman needs only to regulate the inflow of juice and steam-pressure on the first pan. It is perhaps needless to state that a very uniform vacuum in the third pan is requisite to satisfactory work with these circulators.

Owing to the rapidity of the evaporation in multiple-effects there is more or less entrainment of juice with the vapors. This juice is recovered by leading the vapors against baffle-plates, usually in a greatly enlarged section of the vapor-pipe. The enlargement of the pipe reduces the rate of the travel of the vapors and permits the deposition of the entrained juice. This device is called a "save-all" or "catch all."

An evaporator having two pans is a double effect, three pans a triple effect, four pans a quadruple effect, etc. The principle in all types is the same as that outlined for the standard triple effect. With higher combinations than three pans the first pan is worked under pressure instead of vacuum and is known as the "pressure-pan."

**80. Incondensable Gases.**—The calandrias of multiple effects are provided with pipes for the removal of the incondensable gases. A careful supervision of the removal of these gases is necessary, not only because they reduce the efficiency of the heating-surfaces, but, further, they frequently destroy them. The gases contain ammonia derived largely from the decomposition of the amids of the juice, and partly from other nitrogenous constituents. The ammonia should be removed, since it attacks the upper ends of the copper

tubes in the steam-space and ruins them. Juices from new canes contain more ammonia than those from old canes.

**81. Steam Economies in Evaporators.**—It has long been recognized in the beet industry that the greatest possibilities for steam economies lay in making some of the vapors from the evaporators do double or triple duty in other forms of heating. These possibilities are now being made use of to a considerable extent in the cane industry, the means used being as follows:

*"Robbing" the First Effect.*—More steam is generated in the first and second bodies of a quadruple effect than is needed by the evaporation in the others, and this "extra steam" is "robbed" or taken off for use in juice heating. It is general practice, however, to provide the first vessel with a larger heating surface (frequently double) than the other vessels in order that all the juice may be thus heated. This use of the vapors extends double effect heating to the juices, and sometimes to other forms of heating also.

*Vapor Cells.*—An extension of the above idea is the installation of an additional vessel ahead of the evaporator, to the calandria of which exhaust steam is added, while all the vapors are withdrawn from it for juice heating. Pressures in the various bodies will vary with changes in conditions, but the following, observed in such a system in operation, will serve as an illustration: Vapor cell—calandria, 11 pounds; vapor space 7½ pounds. First body—calandria, 8 pounds pressure; vapor space, 5 pounds pressure. Second body—1-in. vacuum; third body—14-in. vacuum; fourth body—26-in. vacuum. The saving in fuel, where all juice is heated by vapors from the evaporating system, is close to 5 per cent of the total consumed by the factory. Juice heaters for use with these methods are usually the closed return-tube type and must be made larger than for use with exhaust steam because of the lower temperature of the vapors doing the heating.

*Double-effect Vapor Cell.*—A further extension of the vapor cell is the dead-end double effect, exhaust steam being supplied to the first calandria while the vapors from the second body are all sent to the juice heaters. The partly concentrated juice goes on to the multiple effect to be further evaporated to 60° Brix. This system is said to save 8½ per cent of the steam required by a factory using a straight quadruple<sup>2</sup> and the total evaporator heating surface is no greater. Dead-end triple-effect vapor cells are also being tried.

*"Pauly-Greiner" Pre-evaporators.*—These have been used extensively in the beet industry and have come into use recently in many modern factories where electrification and other means of steam economies have resulted in a marked reduction in the quantity of exhaust steam. These "Pauly's," as they are termed in Cuba, are similar to the bodies of a standard effect, except that live steam (35 to 40 pounds pressure) is admitted to the calandria and the resulting vapors from the juice go directly into the exhaust steam system of the factory. The amount of live steam admitted to the calandria is so regulated (frequently automatically) that a constant pressure is maintained in the exhaust main. Such a pre-evaporator can only be an economy where the shortage of exhaust steam is sufficient to justify its installation.

The live steam gives a high rate of evaporation and therefore a great

<sup>2</sup> G. W. Connon. Facts About Sugar, April 30, 1927.



increase in the capacity of the evaporator equipment, and each pound of steam that can be sent to the exhaust system represents a saving of nearly a quarter of a pound of steam in the total amount required to operate the factory.

The "regenerative" pre-evaporator which has been installed in one or two Cuban factories consists of the type already described connected with a series of "thermo-compressors" (Fig. 27). A part of the vapor goes direct to the exhaust system while the remaining part goes to the thermo-compressors in which the low pressure steam is compressed to a higher pressure by means of the velocity of a jet of live steam much on the same principle as a steam injector. The steam so compressed is sent back into the calandria of the pre-

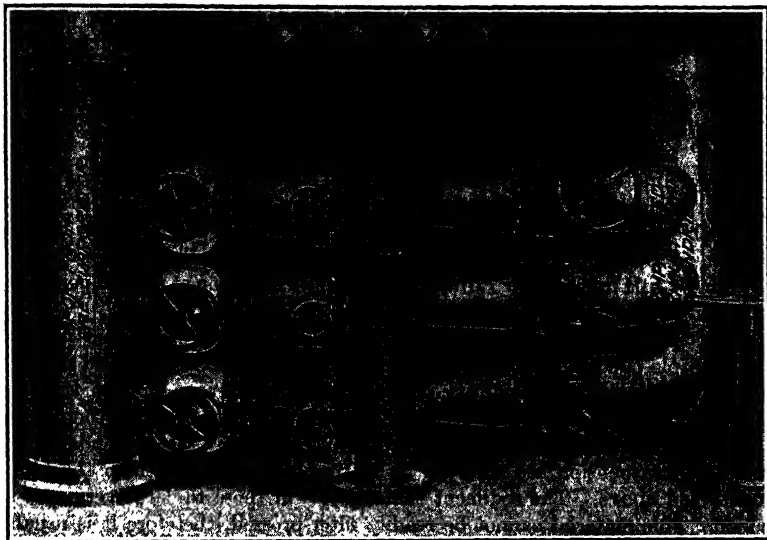


Fig. 27.—Thermo-Compressors Connected to Evaporator.

evaporator, each pound of live steam thus being made to do double duty. This system is only of value where exhaust steam is deficient for boiling purposes.

The Honolulu Iron Works describes an installation of this type as consisting of "a standard quadruple effect evaporator having 12 feet 6 inches diameter cells to which was added a regenerative pre-evaporator 14 feet 6 inches diameter, the pre-evaporator being designed to supply its own thermo-compressors with approximately 45,000 pounds of vapor per hour, as well as to furnish 45,000 pounds of exhaust steam at 10 pounds pressure to the house exhaust mains. This work was accomplished by the use of 45,000 to 50,000 pounds of live steam per hour, giving approximately the economy of a double effect in a single cell evaporator. The completed combination increased the capacity of the evaporator about 30 per cent."

Evaporating juice at 10 or 12 pounds pressure (239°-245° F.) was studied by Noël Deerr in his investigation of the effect of high temperatures upon cane sugar in solution. Among other conclusions he arrived at the following: (1) "The system obtaining in cane-juices is a very complex one, consisting of very variable amounts of salts of both strong and weak acids, and of free alkali. Hence a temperature that may be safe with one juice may cause serious inversion with another. With conditions usually prevailing in local factories (Hawaiian Islands), juices should suffer a half hour's heating at 120° C. (248° F.) with no detectable loss of sugar. It would be conservative to adopt this temperature as the highest to which cane-juice should be subjected during evaporation, though under a careful system of control and observation a temperature of 125° C. (257° F.), or even 130° C. (266° F.) for shorter periods, might be permissible." (2) "The sterilization of cane-sugar products is possible since it occurs almost instantaneously at 125° C." (3) "The use of high-temperature evaporation and the preheater system of evaporation and also the sterilization of all cane-sugar house products is possible under a rational control."

Unfortunately the determination of pH in sugar house products was not in use at the time Deerr reached these conclusions. It is possible that inversion takes place at these temperatures at reactions somewhat above 7.0 pH though agreement on this point has not been reached by investigators. Certainly acid juices (pH much below 7.0) will show definite inversion at the temperatures maintained in the Pauly pre-evaporator.

**82. Settling the Sirup.**—After the concentration of the juice to a sirup in the multiple-effect evaporators, it is pumped to combined settling- and storage-tanks. A considerable quantity of impurities that were soluble in the thin juice are insoluble in the sirup. A part of these impurities deposit themselves upon the heating-surface of the evaporator, forming a hard scale. Those which remain in suspension are removed by settling and decantation in the tanks mentioned above. The sediment that collects in these tanks is very rich in sugar. This material cannot be readily filter-pressed, therefore it is usually pumped to the defecators or clarifiers, to be diluted with juice and the precipitate removed by decantation. Large modern factories have little time for settling the sirup.

Instead of pumping the sirup directly to the settling-tanks, it is often first boiled and skimmed, *i.e.*, clarified. This is probably beneficial in the manufacture of white sugar, but at the expense of sucrose, through inversion. The glucose-ratio of the sirup rapidly increases during the clarification, and the scum and foam that rise to the surface have approximately the same coefficient of purity as the sirup from which they are derived.

**83. Evaporator Scale.**—The scale which forms on the tubes of the evaporators must be removed at frequent intervals as it is a very poor conductor of heat. Its composition varies somewhat with that of the cane and in some localities forms very much faster than in others. The tubes of the first pan, which always contains thin juice, scale little compared with those of the other pans. The third pan of a triple-effect contains the thickest and most obstinate scale.

The following analyses of the scales indicate their composition in the different pans of a quadruple effect: <sup>3</sup>

	1st Pan, Per Cent	2d Pan, Per Cent	3d Pan, Per Cent	4th Pan, Per Cent
Phosphate of lime.....	57.85	56.98	15.02	7.49
Sulphate of lime.....	2.02	1.92	0.54	1.65
Carbonate of lime.....	3.25	4.68	19.55	9.93
Silicate of lime.....	7.86	13.31	0.71	7.02
Oxalate of lime.....	.....	.....	11.32	11.27
Iron oxide.....	2.03	1.53	2.31	2.58
Silica.....	7.79	7.43	39.26	54.34
Combustible matter.....	20.37	13.41	11.04	5.08

**84. Cleaning Evaporator Tubes.**—The tubes may be cleaned by moistening the scale with water and scraping the surfaces. This is a very laborious and tedious method, therefore the following is usually used, and applies also in cleaning the coils of the vacuum-pans. The pans are filled to above the level of the tubes with a caustic soda solution containing from 1 to 2 pounds of the soda per cubic foot of solution. This soda solution is boiled during several hours and is then run from the pans into an iron or lead-lined store-tank. The pans are washed with water and then boiled with dilute muriatic acid, which is afterwards run into the sewer. The acid should be strong enough to be very acid to the taste. This treatment if repeated at intervals of about a week will usually keep the heating-surface in good condition. At the end of the manufacturing season it is usually necessary to give the tubes a thorough scraping.

The alkali solution may be used repeatedly, adding caustic soda from time to time as it becomes weakened. The soda solution is always boiled under atmospheric pressure in cleaning evaporators and pans.

In Hawaii a method of spraying the tubes with caustic soda, soda ash, or a mixture of both, has been favorably reported on.<sup>4</sup> Soda solution of 30° B $\acute{e}$  is pumped into the bodies of the evaporator through ordinary garden spray nozzles placed so that the solution falls in a fine rain over the tubes. A small amount of steam in the calandria serves to keep the soda hot. The soda drains back to the supply tank and the spraying continues for about two hours, after which the effect is washed with water. Acid is used subsequently if needed.

<sup>3</sup> Prinsen-Geerligs Kobus Archief., 1900, 694.

<sup>4</sup> N. E. Wright, *The Planter*, Vol. 79 (1927), No. 23.

## CHAPTER X

### PRESERVATION OF THE JUICE AND SIRUP DURING TEMPORARY SUSPENSION OF MANUFACTURE

**85. Storing of Juice and Sirup during Shut-downs.**—It may be necessary in the event of the breakage of the machinery or delay from other causes to store juice and sirup several hours, or even days. Sirup will usually keep sweet, under tropical conditions, from 36 to 48 hours without the use of a preservative, provided that it is of fully 54.3° Brix (30° Bé) density; is stored in clean tanks, not in the vicinity of fermenting sugar solutions and the pH is 6.8 or above. Raw juice will keep sweet but a very short time without a preservative and in the event of a shut-down all juice should be limed, heated and sent to the defecators. The method of manufacture influences the keeping qualities of juices and sirup, those treated by the sulphur processes remaining sweet much longer than others prepared with lime only.

The length of time which defecated juice will keep without preservatives depends on the temperature at which it is stored and the reaction of the juice. McAllep<sup>1</sup> reports that juice limed to a pH between 7.6 and 8.3 (tested after heating) and held at a temperature of 180° has frequently been kept for twenty-two hours with no acidity developing sufficient to cause inversion of sucrose. H. S. Walker is reported by McAllep<sup>2</sup> as keeping juices over a twenty-four or thirty-six-hour shut-down without preservatives by reducing the temperature of the stored juices to 180° F. and not permitting it to fall below 160° F. (below which point bacterial action will develop).

**86. Preservation with Formaldehyde.**—Formalin, which is a 40-per cent solution of formaldehyde in water, is a cheap and effective preservative for juices and sirups during shut-downs. Its use was first advocated by Dr. Spencer in Cuba in 1908 and has now become quite general.

Clarified juice, made by the sulphur process, may be stored in clean tanks for thirty-six hours and probably longer by the addition of 3 cc. of a 40 per cent solution of formaldehyde per cubic foot of juice (about 1 part formalin to 10,000 parts of juice). Juice clarified by the ordinary process requires considerably more than this quantity of formaldehyde, usually from 6 to 8 cc. (from 1 : 5000 to 1 : 4000).

Spencer preserved 30° Baumé sirup during a period of seven days by the addition of 6 cc. of 40 per cent formaldehyde solution per cubic foot. Possibly less formaldehyde would have answered, but it was not deemed advisable to incur risk. The capacity of the storage-tanks was 5000 gallons each.

<sup>1</sup> The Planter. Vol. 72 (1924), No. 6.

<sup>2</sup> *Loc. cit.*

Juice may be preserved twenty-four hours in the defecators, without removing the scum or mud, by the use of formaldehyde. The measured quantity of the preservative should be mixed with the juice immediately on starting to fill the defecator. With closed separators of the Dorr type the formaldehyde should be added to the pump tank and pumped through the heaters to make certain of thorough mixing.

Dr. Spencer found that there was always a slight deterioration of juice even in the presence of formaldehyde. This was undoubtedly due to the high temperatures at which the juices were carried, and can be entirely prevented by maintaining a temperature not higher than 180° F. on any juice carried over, as reported by McAllep and Walker in the work cited above. Insulation with dry bagasse of tanks used for storage of juices to keep the temperature from dropping below 160° F. is advocated by McAllep.

It has been shown by Norris<sup>3</sup> and Meade<sup>4</sup> and corroborated by Owen bacteriologically that formaldehyde in too small amounts (say 1 : 50,000 or 1 : 100,000) will actually increase the deterioration due to micro-organisms, so no attempt should be made to economize on the preservative. The cost of formaldehyde in barrels is so small that it would usually be advisable to employ it in even larger quantities than is indicated above. It practically all disappears from the juice in the evaporation.

<sup>3</sup> Hawaiian Bulletin, No. 23.

<sup>4</sup> Proc. VIII Int. Cong. Applied Chem., N. Y. 1912.

## CHAPTER XI

### CRYSTALLIZATION OF THE SUGAR

87. **The Vacuum-pan.**—The sirup obtained in the concentration of the juice still contains from 35 to 45 per cent of water. The content of water could have been largely reduced in the multiple-effect (assuming sufficient evaporator

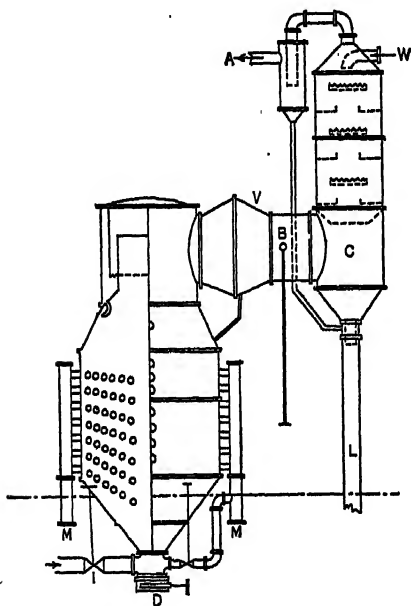


FIG. 28.—Coil Pan and Condenser.

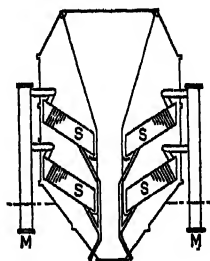


FIG. 29.—Double Calandria Pan.

capacity) but to facilitate the graining of the sugar further evaporation is conducted in a vacuum-pan in single-effect. Formerly, sirup was always turned out of the evaporators at 54 Brix or lower but recently many factories have increased the sirup density to 60 or even 65 Brix in order to make use of as much steam as possible in the multiple-effect for the sake of fuel economy.

Modern vacuum-pans are of two types—coil and calandria pans. The older style is the coil pan (Fig. 28) which is a vertical cylindrical vessel of cast iron with a conical bottom; fitted as shown with a number of copper steam-coils in which live steam (generally reduced in pressure to 40 or 50 pounds) is circulated.

It has a large door or strike-valve at the bottom, for the discharge of the sugar. The roof or dome of the pan is connected with a save-all and condenser by a large vapor-pipe and with a vacuum-pump by a small pipe. The vapor-pipe is usually made very large, that the velocity of the vapors may be comparatively slow, thus reducing the entrainment of sirup. The pan is also provided with eyeglasses or lunettes through which to observe the progress of the boiling liquor, and a proof-stick for the removal of test samples. The apparatus is so arranged that the panman can vary the temperature of the boiling liquor by increasing or decreasing the vacuum which is produced by the pump and condenser.

The calandria pan differs from the coil pan only in the character of the heating surface. The one shown in cross-section in Fig. 29 is a double calandria of the "basket" type, so-called because the calandrias (*S*) slope toward the large center down-take pipe to facilitate discharging the massecuite from the pan and the draining of the condensed water from the steam space. Flat calandrias have no slope, as the name implies, and are frequently employed where coil pans have been changed to the calandria type. Calandria pans are generally operated on exhaust steam at 10 pounds pressure.

The large number of short copper tubes makes it possible to carry much greater heating surface (30 to 50 per cent more) than in a coil pan of similar size, and the pan is left freer from obstructions to circulation, both of which characteristics combine to make it possible to boil a strike in a calandria pan in a shorter time than in a coil pan, despite the higher steam pressure used in the latter. Coil-pans have been gradually replaced by the calandria type and some of the latest design cane-factories have no coil-pans at all.

The vapors from the pan (of whatever type) are condensed in a special condenser (*C*) and flow with the condensing-water through a torricellian tube or "leg-pipe" *L* (see Fig. 28) to a hot well and thence from the factory, while the air is led off in a separate pipe and removed by the pump. In many tropical factories there is a shortage of water and the condenser-water is run to cooling towers or spray ponds for cooling and returned to the factory for re-use in the condensers.

**88. Boiling Sugar to Grain.**—In order to facilitate the description of pan-boiling, and to familiarize the chemist with factory terms, a few expressions used by sugar-boilers will be first given. These expressions differ somewhat in the cane, beet, and refining branches of the industry. Only those customary in cane-work will be given:

The concentrated juice is called "sirup" or "meladura," the latter word is Spanish and is used by foreigners in Cuba and other parts of Spanish-America. The concentrated mass of crystals and molasses form the "massecuite," or when boiled partly with sirup and partly with molasses, "mixed massecuite," and each boiling is called a "strike." When a portion of a strike is removed from the pan and the remainder is left as a footing or nucleus upon which to boil another strike, the portion so left is termed a "cut." There is some confusion in regard to the use of the word "cut," as many sugar-boilers use this word for the massecuite removed from the pan. The process is termed "cutting."

Where two or more pans are worked side by side they are often connected with one another by large pipes, with suitable valves, for drawing the massecuite from one to another. These are called "cut-over pipes."

In modern practice, when a grained strike reaches a certain stage, diluted molasses is drawn into the pan instead of sirup and it is said to be "boiled in," or "boiled in on grain-sugar."

When the liquor in the pan has been concentrated to a certain density and is more or less saturated with sugar, it is said to have reached "proof." The proof may be "strong" or "weak" as the liquid is of greater or less density. This expression is still further modified, as will be explained in connection with the boiling of molasses-sugars.

The process of boiling sugar is as follows: The pan is closed, the vacuum pump is started, and when the gauge shows a vacuum of 15 to 20 inches of mercury, the charge-valve is opened and sufficient sirup is drawn into the pan to cover several of the coils. Steam is turned into the coils that are covered with sirup and the sirup is rapidly concentrated to proof. The quantity of sirup used depends upon whether the boiler wishes "to grain" high or low in the pan. In making fine-grained sugar he will grain high, as he needs many crystals and comparatively little room for their growth; on the contrary, in making coarse-grained sugar he will grain low, and form few crystals.

The procedure of graining in a calandria pan is much the same, the steam being turned on only in the calandria that is covered with sirup or massecuite, since it is evident that steam pressure may not be maintained in any heating surface not covered with liquid without burning the adhering sugar.

As the liquor boils down, the steam-valves of the upper coils are successively closed, and as it becomes more and more concentrated, the portions projected against the eyeglasses flow more and more slowly and the panman increases or decreases the quantity of water injected into the condenser until the liquor boils at the desired temperature. At this stage there are two methods of procedure: (1) The concentration is continued until the liquor is sufficiently supersaturated with sugar, as is indicated by a sample drawn by the proof-stick. The water of injection into the condenser is now increased and the steam-pressure on the coils reduced, thus cooling the boiling mass, increasing the degree of saturation, and forcing minute crystals of sugar to form. These manipulations vary somewhat according to the vacuum-pan itself and the quality of the sirup, but in general are as described. (2) The second method of graining is that usually employed in the United States. The concentration of the liquor is continued until crystals of sugar separate, and when the panman considers that there are sufficient crystals he raises the temperature of the pan and injects more sirup. The first method produces a sugar of more uniform grain than the second, since all of the crystals required are formed at once. (3) A method introduced by Zitkowski in beet sugar work which has rapidly gained favor in refining and cane-factory sugar-boiling consists of boiling down to supersaturation as in method (1) and then drawing into the pan a small quantity (a pound or so) of sugar dust. It was believed at first that this dust formed the nucleus of the grain formed by its introduction, but it is now agreed that it serves merely as a "shock" to the super-



saturated sugar solution and that the crystals are formed accordingly. A very regular grain results from this method properly employed. The use of a microscope of 50 diameters magnification is advocated for pan-boilers by technologists in the Hawaiian Islands.

After the crystals are formed, sirup is drawn into the pan from time to time as the water evaporates. This charging of the pan with sirup, and judging just when to charge, requires much skill and practice on the part of the boiler in order to secure the best results. If he injects sirup when the mass is too concentrated, with the liquor surrounding the crystals too rich in sugar, fresh crystals will be formed and must be remelted. These crystals are termed "false grain," and where formed at a late stage through carelessness of the sugar-boiler and are not removed, they impede or even prevent the curing of the sugar in the centrifugals. False grain may be formed by charging the pan too freely with sirup, or by cooling the massecuite while the sirup surrounding the crystals is rich in sugar and of such density that it is supersaturated at a lower temperature. Also, if a sufficient number of crystals have not been formed when graining the strike, there is great risk of the formation of false grain, after the crystals have grown to large size. If the false grain is formed and not removed at this stage of the boiling, the sugar cannot be purged of molasses. False grain is gotten rid of by raising the temperature of the pan and drawing in additional sirup to melt the fine crystals.

As already stated, as the water is evaporated, sirup is drawn into the pan from time to time until the strike is finished. The massecuite is kept very free, *i.e.*, of comparatively low density, until the crystals are large or the pan is about two-thirds filled. At this stage it is gradually boiled dryer and dryer until near the end of the operation, thus impoverishing the molasses surrounding the crystals, and finally the massecuite is evaporated to an apparent degree Brix of about 93, and is then discharged from the pan. The massecuite should be as cold as possible at this stage.

There are various methods of estimating the proper moment for striking the massecuite, *i.e.*, discharging it from the pan. This is usually determined by withdrawing a sample with the proof-stick and forming it into a conical heap upon the thumb with the finger. The consistency of this massecuite, as shown by its appearance, rate of flattening, etc., are noted by the sugar-boiler, and a practiced man can in this way judge within narrow limits when to drop the strike. A refractometer, directly attached to the pan, is used in Europe to determine the correct time to discharge the pan, as well as to regulate the intake of sirup during the boiling process. This procedure has not yet been adopted on this side of the water.

During the boiling, care must be observed after each charge of sirup to concentrate the mass sufficiently to force the sugar to deposit itself upon the crystals already present and not to form new ones. Unnecessary concentration causes a waste of time and steam.

A method of charging a pan with sirup, employed with many vacuum-pans, but not applicable with all, is by means of a "set feed." After the formation of the grain the charge-valve is opened and so adjusted that the flow of sirup into the pan just compensates for the evaporation. The opening

of the valve is adjusted from time to time as the concentration varies, with variations in the steam-pressure, the density of the sirup, etc. This method of charging, with the first method of graining, produces a very uniform quality of sugar, and a good yield from the massecuite. An irregular grain, with many minute crystals, from false grain that has not been melted, results in a low yield of sugar from the massecuite.

In boiling a massecuite so as to obtain the maximum yield of sugar from it, the full number of crystals required should be formed at graining, and the crystals should be of moderate and very regular size. Of course, false grain at any stage of the operation must be avoided. The formation of too few crystals results in rich molasses, since there is not sufficient crystal-surface to take up the sugar, and besides increases the risk of the formation of false grain. When boiling a strike on a very small nucleus of massecuite or on a small cut, there is also risk of false grain or of a partially exhausted molasses.

The temperature to be maintained in the massecuite during boiling varies with the desired grade of sugar, whether hard or soft, white or raw. American practice also differs somewhat from that of Java in respect to temperatures. The Java superintendent prefers relatively low temperatures. As will be noted in the chapter on sugar-refining, page 177, hard sugars are boiled as high as 180° F. in the American refineries, with no inversion. Somewhat lower temperatures are customary in the white-sugar factories of Louisiana.

High-test raw sugars should be grained at temperatures approximating 160° F., and the high temperature should be continued until the grain is well developed. The temperature may be lowered in the latter part of the strike to promote reduction of the purity of the molasses surrounding the crystals. Massecuites that are to be further crystallized either in motion (crystallizer massecuite) or at rest in tanks should be boiled at as low temperature as possible. By keeping the temperature low (150° or lower) foaming and swelling of the massecuite in the crystallizers are reduced to a minimum.

**89. Methods of Sugar Boiling.**—Since the last edition of this book was written the progressive sugar countries have seen the complete adoption of those radical changes in sugar-boiling methods and the handling of massecuites which were described as in the transition stage at that time.<sup>1</sup> These improvements include: (1) the production of one grade of raw sugar only; (2) the almost universal use of "seed grain" in the higher test massecuites; (3) the adoption of crystallization-in-motion machinery and (4) the discontinuance of "string-proof" boiling and the use of the "hot-room."

The disappearance of "molasses" sugars from the market (Sec. 157) and the production of "centrifugal" sugar only has been attended also by a steady increase in the test of the raw produced. Where 96° polarization was the aim

<sup>1</sup> Dr. Spencer was largely responsible for the development of these improvements, particularly in Cuba and Porto Rico. He introduced the use of seed made from "grain" strikes; also the double-purge method of purifying this seed; he was the first to make a single high-grade product of uniform composition, and the boiling methods originated by him were so practicable that they spread rapidly, resulting in the fulfillment of his prediction made in 1910 that "within a few years the string sugars will almost cease to be produced." (G. P. M.)

fifteen years ago, 96.5° to 97.0° is common in Cuba today, while the Hawaiian Islands are regularly producing raws of 97.5 to 98.0. Pan-boiling systems have been modified so as to give these higher polarizations, and the various methods to be described here should be judged from several viewpoints; (1) the refining quality of the sugar produced; (2) its keeping quality; (3) the purity of the final molasses; (4) the amount of re-boiling required (cubic feet of massecuite per 100 pounds sugar produced) and (5) the flexibility and simplicity of the system as a factory operation.

*Pan Room Definitions.*—Raw sugar manufacturers often designate their strikes by the letters "A," "B," "C," etc., or number them to distinguish the grade of massecuite and not the pans themselves. Lettering of massecuites is used as a designation more often in Hawaii and Java; numbering in Cuba. In refinery work, the pans are lettered and the same pan is usually employed for the same grade of sugar because of its proximity to a certain mixer and set of centrifugals, but this is not so generally true in a factory.

With the complications introduced into pan practice by boiling-in molasses many find it simpler to designate massecuites by names rather than numbers. Where the first strike is boiled with pure cane-sirups, the second with first molasses, the third with second molasses, etc., the strikes may be numbered "first," "second," and "third," but where all the pans are boiled in part with molasses there is some confusion in the nomenclature. The various names and numbers used will be given in describing the different methods of pan-boiling.

The boiling-back of molasses has been practiced in Louisiana for nearly fifty years, especially in making grocery-sugars. The mixed massecuite was often termed "grain seconds." With the advent of crystallizers the present methods of boiling were developed simultaneously in Louisiana, Cuba, and Java. The work in the East Indies was apparently based entirely upon European methods, and that in Cuba upon Louisiana and beet-sugar methods.

In describing the different pan-boiling systems it will be understood that purities given are average figures only which will vary through fairly wide ranges depending upon the purity of the original sirup, the equipment of the factory and other considerations. The purities of the various massecuites are generally arranged with a view to giving a fixed purity to the molasses that goes to make up the bulk of the crystallizer massecuite. A purity of 52° is a convenient number for this molasses.

The purity to be given the final or crystallizer massecuite depends upon several factors, viz., vacuum-pan capacity, crystallizer and centrifugal capacity and the desired exhaustion of the final molasses. The lower the purity of this massecuite the longer is the time required in boiling the strike. A very low purity massecuite must remain a long enough time in the crystallizers in order to reduce its viscosity by crystallization of the sugar and promote rapid purging. Where molasses is sold upon a basis of a certain sugar content, this also must be considered in fixing the purity of the second massecuite. For obtaining final molasses of apparent purities below 30°, early in the season in the Tropics, and from 30° to 35° when the cane is ripe and of high purity, the massecuite should be reduced to below 60° purity. Where the pan, crystallizer

and centrifugal capacities are large, the purity may be reduced to 56° or even lower with correspondingly lower final molasses purity.

The first and second massecuites may be purged immediately after leaving the vacuum-pan, and should produce the same grade of sugar and molasses of widely different purities. The second or crystallizer massecuite is purged after remaining three or four days in the crystallizers. This massecuite yields a sugar that was formerly sold as seconds but is now converted into sugar nearly equal to the firsts by double purging.

It should be noted that in boiling-in molasses this should be accomplished after no more sirup is required by the strike. The molasses must be free of sugar-crystals and should be warmer than the massecuite in the pan. It should be reduced to a density of about 54° Brix, at pan temperature.

*Massecuites Boiled with "Seed."*—Grain, mingled into a magma with some convenient vehicle such as sirup or molasses, upon which to build other grain is popularly called "seed." Its use is general no matter what system of boiling is employed. The evolution of this practice is typical of the efforts that are being constantly put forth in modern sugar-making to produce a raw of better quality. The earliest form of the method was in Java and the Hawaiian Islands where the strike was started upon a footing of magma formed of string-sugar mingled with sirup. A sugar of good grain and test may be made in this way, and the factory may produce a single grade of product without remelting the string-sugar, but the objectionable feature is that the nucleus of the raw sugar crystal (the string-sugar) is of low test, and sugar so made will not give a high purity "washed-sugar" in the affination process of the refinery (Sec. 158).

The next modification (introduced by Dr. Spencer in Cuba), differed only in the source of the seed. The grain-sugar obtained from the crystallizer strikes was mingled with first or second molasses to form a magma and this was used as a footing for starting first massecuites or mixed strikes. It was soon recognized that the use of molasses for mingling the seed introduced color and impurities into the higher massecuites, hence sirup is now used for mingling. Another advance (also introduced by Spencer) is the "double-purging" of the sugar used for seed. The sugar purged from crystallizer strikes is mingled with a low-purity molasses and sent to a separate set of centrifugals where it is repurged, washed and then mingled with sirup (meladura) for use as seed in high-grade strikes (see Sec. 136). Seed sugar, repurged in this way, is practically of the same test as the grain which builds upon it so that the resulting raw is uniform throughout its crystal structure. Magma for seed is stored in a crystallizer in motion and is always available without loss of time and in this respect is preferable to cutting massecuite from another pan.

*Two-massecuite Method.*—A flow diagram of this method is shown in Fig. 30. All the massecuites from which commercial raw sugar is to be produced are reduced to the same purity (from 70 to 74°) with molasses from previous strikes so as to give a second molasses of about 52° or lower, which is boiled back into a massecuite of 56° formed on grain made from sirup. This massecuite goes to crystallizers (as do all final massecuites in modern practice) where it remains several days, after which it is separated into sugar and final molasses in the

centrifugals. The sugar from the crystallizer strikes is mingled with molasses and taken back for seed into the commercial sugar strikes.

The two-boiling method has only the advantage of simplicity to commend it. The disadvantages are that all the output of sugar is from relatively low test massecuite, with a detrimental effect on refining and keeping quality; also, due to reboiling of so much molasses, about 15 to 20 per cent more massecuite per 100 pounds sugar produced must be boiled, putting that much extra work on pans, crystallizers, and centrifugals, with increased fuel consumption. For these reasons the method has been entirely discontinued in Hawaii, where it was formerly very general, and is in use in only a few factories in Cuba.

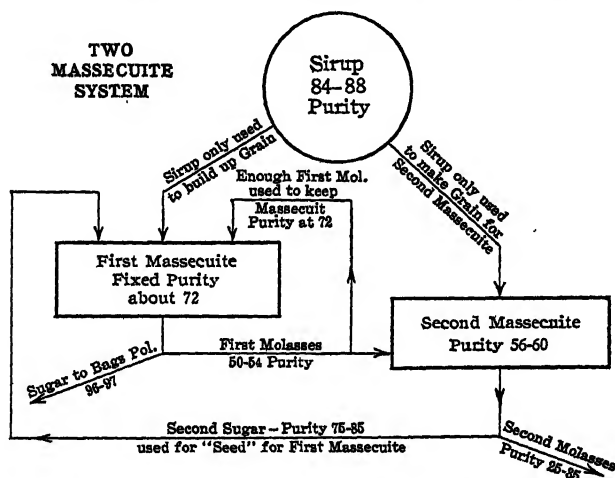


FIG. 30.—Flow Diagram of Two Massecuite System.

*Three-massecuite Method.*—This is the method used (Fig. 31), with various modifications, by most of the Cuban houses. A first massecuite is boiled from sirup and first molasses to a purity of 80–83; a second massecuite, frequently called a “mixed massecuite,” is boiled from sirup and first molasses to about 70 or 72 purity; and a third massecuite, known as “second” or “crystallizer” massecuite (where the term “mixed” is used for the second boiling), is made at about 56° purity. In actual practice the first massecuite is generally boiled on seed grain from the crystallizer strikes, mingled with sirup; while the “mixed” strikes are boiled on seed mingled with first molasses, or on a footing from a cut from a first massecuite. The crystallizer massecuites are usually started on a footing cut over from a first massecuite pan.

The sugar from the first and second massecuites are purged immediately after leaving the vacuum-pan and should produce the same grade of sugar. It may be necessary to wash the sugar from the second (mixed) massecuite to accomplish this. The crystallizer massecuite is purged after remaining in the crystallizers three or four days, and yields final molasses and a sugar which is

used for seed. This sugar may be mingled with molasses, repurged and washed to give a sugar practically equal to the first sugars, in which form it may be used as seed or sold directly, though the latter is not frequently done.

The three-masseccuite method, when double-purged crystallizer sugar mingled with sirup is used for seed, is subject to one criticism only. If the purity of the original sirup is above the desired purity of the first masseccuite, the amount of first molasses used to reduce the masseccuite purity entails just that much extra boiling, which may amount to from 5 per cent to 8 per cent of extra pan- and centrifugal-work. With high sirup purities ( $86^{\circ}$  to  $88^{\circ}$ ), such as are met with in tropical practice, this is generally the case. Where double-purging is not used, or where molasses instead of sirup is used for min-

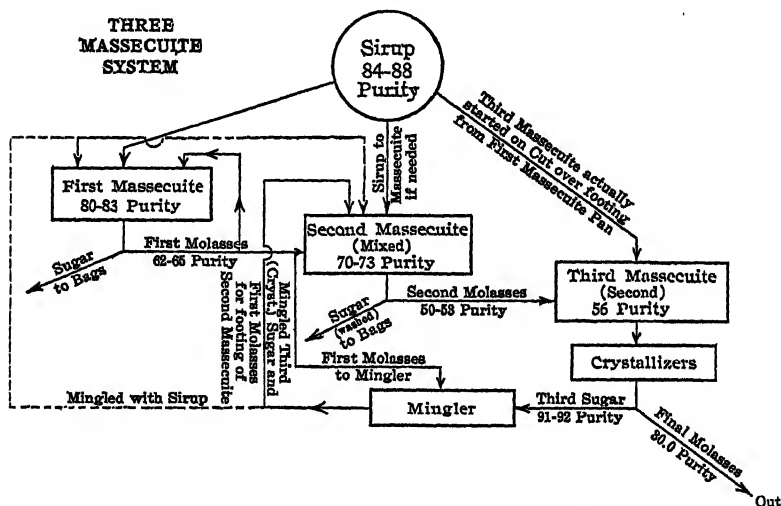


FIG. 31.—Flow Diagram of Three Masseccuite System.

gling the seed, the objections already referred to are valid; viz., that impurities from the low-grade strikes are introduced into the high-grade strikes.

*Four-masseccuite Method.*—This is shown diagrammatically in Fig. 32 and consists of using the "natural drop" in purities without boiling any lower-purity molasses back into higher strikes. (This principle of never reversing the downward tendency of the stream of impurities has been the basis of refinery and beet-house boiling systems for many years.) The first strike is started on seed mingled with sirup and completed with sirup only; the second and third strikes are started on similar seed and then take in all the molasses of the preceding strike, while the crystallizer strikes are all boiled on a footing of sirup *boiled to grain* (not from seed). The figures as shown in the diagram are for an original sirup purity of  $86^{\circ}$ , but it is evident that with a change in this purity the other purities will change accordingly, the only predetermined purity being that of the crystallizer strike which is held at approximately  $56^{\circ}$ .

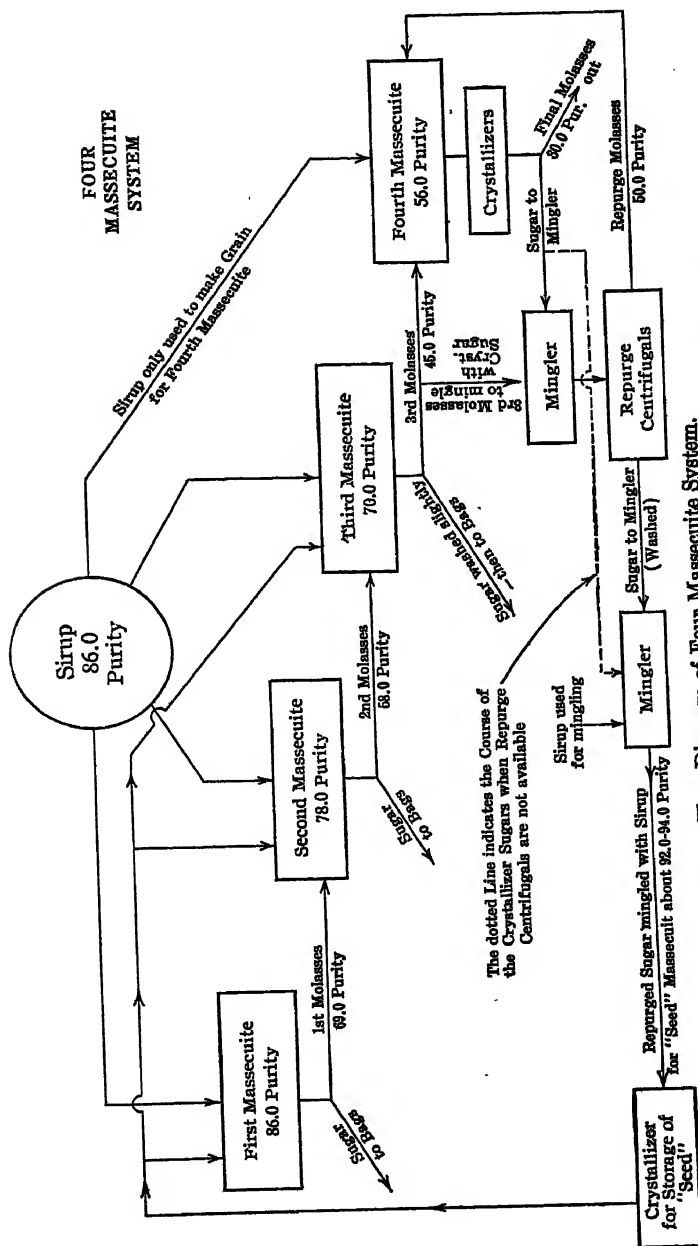


FIG. 32.—Flow Diagram of Four Massecuite System.

NOTE.—Purities given are illustrative only. These may vary rather widely in practice.

If the sirup purity approximates 80° or lower, three boilings only are needed to reduce the molasses sufficiently and the system becomes identical with the three-boiling method. With unusually high sirup purities (say 90° or above) five boilings may be necessary to accomplish the reduction of the molasses to the proper degree.

The four-massecuite method with double purging of crystallizer sugars for seed was developed in Cuba by Wm. B. Saladin, General Superintendent of the Cuban-American Sugar Co. The resulting sugars are of even composition, and polarize 96.5 to 97.0 without resorting to washing (except for sugars from third massecuities which require a quart or so of water per charge). The system gives a minimum of reboiling, cubic feet of massecuite per 100 pounds sugar produced being lower than for any of the other systems of boiling. The separation of three grades of molasses causes no great difficulty in actual practice.

*Graining Crystallizer Strikes on Undiluted Molasses.*—Walter E. Smith in Hawaii has devised a direct method of graining low-grade strikes for crystallizer work that is described by H. S. Walker <sup>2</sup> as follows:

"A small portion of molasses of 56–58 purity is pumped to a separate tank without dilution or steaming, and reserved for seed. This undiluted molasses is full of microscopic crystals formed mostly by the shock and the cooling as it is thrown off from the sugar in the centrifugals. The balance of the molasses is blown up with steam and diluted as usual. In starting grain enough diluted molasses is drawn in to cover the calandria of the pan, this is concentrated a very little so as not to dissolve the grain to be introduced, but is not concentrated to its own graining point. Undiluted molasses containing 'natural grain' is then drawn in, a very little at a time, until sufficient grain can be seen. The amount of grain required is soon learned by experience. A low-power microscope (50 magnification) is very useful in observing the appearance of the low-grade massecuite at this point.

"After sufficient grain has been taken in, boiling is continued with diluted molasses as usual until the pan is full. It is then generally necessary to cut either to another pan or to a special tank alongside the pan, making two or three strikes from one graining, in order to build up finished massecuities with grains of 0.3 to 0.5 mm. average length. Without cutting, the grain formed by this method is apt to be too small and dries slowly.

"This method of graining low-grade molasses requires a little practice, but is well liked by pan men after they have once mastered it. It has the advantage over graining on sirup that the purity of the low-grade massecuities is not increased and is a closer approach to straight boiling."

This method of graining has never been used in Cuba. It is doubtful whether it would produce as free purging crystallizer strikes as the Cuban method and it would certainly not give as high purity a sugar for seed as boiling on grain made from sirup.

*Boiling to "String-proof."*—This practice has entirely disappeared from modern factories which are always equipped with crystallizers. The method is variously termed boiling to "string-proof," "smooth," "blank," etc., the

<sup>2</sup> The Planter. 80 (1928), No. 117.



molasses so boiled generally being 58 to 60 purity. Carbonate of soda is frequently drawn into the pan with the molasses and apparently with advantage. The soda is preferable to lime.

In boiling molasses to string-proof, it is simply drawn into the pan and boiled down to the proper density. The determination of this density, by the usual tests of a sample drawn to a "string" between the finger and thumb, is a matter requiring much judgment and experience. The required proof varies with the purity of the molasses and the kind of containers used in crystallizing the sugar. In this crystallization at rest, as distinguished from the modern process of crystallization in motion, the crystals formed slowly settle to the bottom of the car or tank, gaining slowly in size, never becoming large. Such sugars range in polarization from 80° to 90°, varying in test with the quality of the molasses and the skill displayed in the pan-boiling. A suitable density for the massecuite is approximately 89° to 90° apparent Brix.

**90. Crystallization in Motion.**—This process was first used in the European beet-industry. Cane-sugar manufacturers were slow in abandoning string-sugar methods, and when crystallizers were first introduced the strikes were boiled blank and run into them. As the name implies, this process consists essentially in keeping the crystals of a massecuite in motion while completing the crystallization. The best results are therefore obtained in pushing the crystallization so far as is practicable in the vacuum-pan and completing it in the crystallizer.

A theory of the process\* that conforms with general experience with cane-sugar is as follows: If the crystals of a massecuite be kept constantly in motion and be brought intimately in contact with the sugar still in solution, this sugar will deposit upon the crystals already present rather than form new ones, provided there is no sudden reduction of temperature to increase the supersaturation. Manifestly it is impossible to produce a stirring device that will move each crystal independently, in the heavy viscous massecuite, to entirely new environment, so that it may be brought into contact with fresh portions of the sugar in supersaturated solution. Further, there is little possible freedom of motion for a crystal in a dense massecuite, since the film of molasses separating it from its neighbors is but a few thousandths of an inch in thickness. The crystal gradually takes on the sugar in its own immediate neighborhood, reducing the density and viscosity of the molasses surrounding it, and in its increase of size it approaches nearer a fresh source of sugar. Great inequality of density cannot long exist, since the stronger solutions near by must mix with the weakened solution by diffusion. The motion of the stirrers of the crystallizer, by rocking the crystals, promotes this diffusion and a uniform fall of temperature in the massecuite. With the fall of temperature, the saturation of the solution resulting from the growth of the crystals is changed to supersaturation and crystallization continues. A point is finally reached when the crystal size ceases to increase. This is due to the viscosity of the medium and not to lack of a supersaturated solution of sugar. When this point is reached, the massecuite should be diluted with water or if preferred

\* "Crystallization of sugar in after-product massecuites," H. Classen, *Int. Sug. J.*, 14, 284.

with dilute molasses. Diffusion is again possible and the crystallization again progresses. The dilution must be repeated from time to time for the best results. Water is preferable to molasses as a diluent since it does not add solids to to massecuite.

It was formerly considered advisable to boil massecuites for crystallizer work to very high density and then reduce the viscosity in the crystallizer with water as described above. Experience has shown that equally good or better reduction of molasses purities is obtained by boiling to moderate densities and not resorting to dilution in the crystallizer. Ninety-three to 93.5 apparent Brix for a 56° massecuite is good practice.

The method of boiling massecuites for crystallizers as described under "methods of sugar-boiling" are almost universal. Obsolete methods, now rarely resorted to, are (1) A grain strike is boiled as is usual and a part of it is discharged from the pan; hot molasses of about 90° Brix is drawn into the pan and is mixed with the massecuite remaining by boiling. When the mixing is completed the strike is discharged into a crystallizer. (2) A strike of mixed massecuite is discharged into a crystallizer and mixed with a string-strike of higher temperature and moderate density that is flowing from an adjacent pan. (3) A magma of molasses and string-sugar is drawn into the pan and heavy molasses is mixed with it as in the first method.

There is always great danger in these methods of forming false grain. There is difficulty in mixing the molasses with the massecuite or magma in these processes.

Among the advantages of crystallization in motion over the older methods, are a saving of labor and floor space, cleanliness, the production of grain-sugars and the ability promptly to liquidate the factory at the close of the manufacturing season. No massecuite need be carried forward to the next season, thus avoiding loss by fermentation.

**91. Laboratory Control of Crystallization in Motion.**—The apparent Brix and the purity coefficient of the massecuite should be determined to control the density of the massecuite and the admixture of molasses. A sample of the massecuite should be purged in the laboratory apparatus immediately after leaving the pan. The molasses from this should be tested for purity. The immediate molasses from a sample of massecuite of 60° purity in the Tropics should have a purity below 40°. A fall of at least 20° from massecuite to molasses may be expected. The further fall in purity to final molasses depends upon the condition of the cane, and will be less in the Tropics than in Louisiana. The Louisiana cane never matures and therefore the initial purity of the juice is never as high as in the Tropics. The total fall in purity in the Tropics is from 55° to 60° and in Louisiana from 50° to 55°. A final apparent purity number of 25° may be expected early in the tropical season while the cane is immature.

**92. Crystallizers.**—The usual crystallizers are of three types, the third of which is now little used in cane-sugar works: (1) Closed crystallizers. This is a horizontal cylindrical vessel fitted with a powerful spiral stirrer. A water-jacket for control of temperature was provided with the original Bock crystallizers. These jackets are now little used in cane-sugar factories. (2) Open crystallizers. These are simply open tanks of half-cylinder cross-section, fitted with stirrers as in the closed type. (3) Vacuum crystallizers.

These are closed crystallizers provided with vacuum-connections and a steam-jacket at the bottom. This crystallizer is in fact a slow-boiling vacuum-pan with forced circulation of the massecuite.

The large extension of present-day factories has led to the discharge of the massecuite from the pans into the crystallizers through pipe-lines. Moderate air-pressure is applied in the pans to force the massecuite through the pipes. The pipe-lines should have an internal diameter of not less than 14 inches, especially if the crystallizers are located far from the pans.

Crystallizers are used to some extent for the storage of first and mixed massecuites pending an opportunity to purge them. From the point of view of the crystallization, it is not general practice to use crystallizers at this stage to further exhaust the molasses, but with the advent of the newer forms of crystallizers described in the paragraphs following it may be found that rapid cooling of the higher-grade massecuites with cooling-coils will give such a rapid



Küby Mfg. Co.

Fig. 33.—La Feuille Rotary Crystallizer.

exhaustion of molasses as to eliminate one or two boilings in the boiling systems already described.

*Crystallizers with Cooling Coils.*—During the last few years crystallizers with cooling coils in them have been developed. There are two forms of these crystallizers, one with stationary coils and the other with rotating coils. Water is circulated through the coils permitting graduated and rapid cooling and consequently more rapid and complete exhaustion of the molasses. Reports<sup>4</sup> show that molasses of three or four degrees lower purity can be secured in about half the time with the crystallizers equipped with cooling coils as compared with the ordinary type. The best practice is to cool the massecuite to 90–95° F. and then warm it up by circulating hot water in the coils to 120° just before sending it to the centrifugals. There is no doubt that the use of this new form of crystallizer will spread rapidly.

*“La Feuille” Rotary Crystallizer.*—This is a recent improved form of crystallizer consisting of a rotating shell (Fig. 33), containing cooling tubes. The

<sup>4</sup> Keller. Proc. 2d Conf. Int. Soc. Sugar Cane Tech., Havana, 1927.

driving arrangement is a worm drive and pinion which acts on a gear ring mounted on the shell. The cooling surface consists of 2-inch tubes extending the full length of the crystallizer parallel to the axis of rotation divided into two or four coils arranged to make the temperature uniform all through the massecuite. The filling and emptying is done through suitable shear gate valves, the crystallizer being stopped for this purpose.

Extensive comparisons made in Java by Honig and Alewyn show the "La Feuille" to be about six times as effective as a standard type crystallizer without cooling coils. The accompanying diagram gives the results of some of these comparisons.

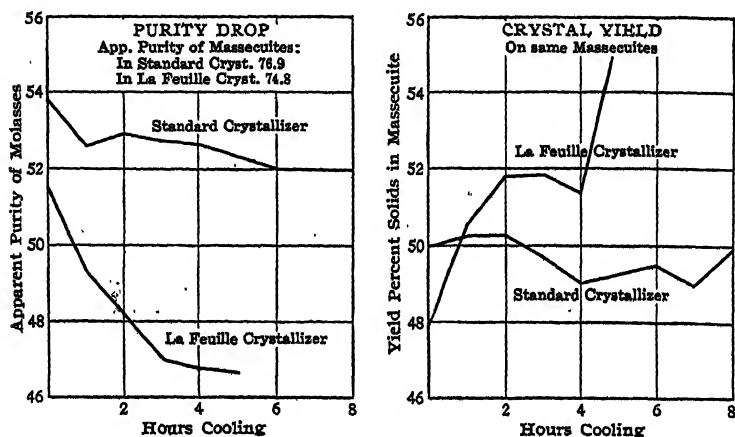


FIG. 34.—Comparison of Operations of the La Feuille and Ordinary Crystallizers.

With these newer type crystallizers the possibility suggested above of eliminating at least one massecuite boiling in the cane-house presents itself, because of the greater drop in molasses purity obtainable from a single boiling.

**93. Solidified Molasses.**—This product seems to be produced only in Java and for shipment to British India for arrack manufacture.

The manufacture of solidified molasses requires no special apparatus. Molasses is first "blown-up" with steam and skimmed and is then simply evaporated to dryness in an ordinary vacuum-pan operating with a very high vacuum. Serious frothing is liable to occur. The proof-stick samples are tested by cooling in water. The cooled sample should have the characteristics described below. The dried molasses, a molten mass, is run into closely woven baskets while it is very hot and solidifies on cooling. The tops of the baskets are covered with burlap for shipment.

The vacuum-pump should be operated for a short time after shutting off the steam from the pan to partially cool the molasses. Cooling by the circulation of water through the coils is also practiced. Gases are produced by decomposition, therefore the pump should not be stopped until ready to break the vacuum.

The usual commercial specifications for this product are as follows: (1) The solid molasses shall break with a clean fracture; (2) it shall sink in water; (3) it shall be too hard to indent with the finger nail.

J. J. Hazewinkel<sup>5</sup> examined solidified molasses to determine whether loss of dry substance occurs during the manufacture. He analyzed the molasses before and after drying, and calculated the results to a basis of the ash content of the original material, assuming it to be a constant. His conclusions were as follows: (1) There is considerable loss of dry substance. (2) The extent of the decomposition is dependent upon the temperature. (3) The reducing sugars in part pass over to organic non-sugar. (4) It is possible that to some extent loss of dry substance is due to decomposition of both reducing-sugar and organic non-sugar. (5) About equal parts of dextrose and levulose are decomposed. In view of the proportions of dextrose and levulose it is probable that polymerization of dextrose to levulose takes place during the long heating.

94. Froth "Fermentation."—When massecuites are boiled at high temperatures they often foam and run over the sides of the containers. The foaming may begin immediately or some hours after striking. This phenomenon is termed "fermentation" or "froth fermentation." It is only in recent years that we have a fairly definite knowledge of the cause of the foam. It is not due to fermentation, that is, to bacterial activity, but to the decomposition of certain salts.

Herzfeld<sup>6</sup> states that Kraisy has recently shown that Lafar's results on the reaction of the invert-sugar with amino acids, *e.g.*, glutanic acid, splitting off carbonic dioxide, is the cause of foaming. Massecuites (sugar-beet) foam suddenly when a temperature of about 65° C. (149° F.) is reached. Kraisy explains the phenomenon by the carbon dioxide remaining in supersaturated solution until the formation of sugar crystals catalyzes its liberation. The viscosity of the massecuite results in foam. Herzfeld also states, always referring to the beet industry, that foaming is rare since the introduction of phenolphthalein for rosolic acid in alkalinity tests. Lime will stop the foaming.

The usual method of stopping foaming in a cane-sugar factory is to pour water upon the surface of the massecuite. This reduces the viscosity and facilitates the escape of the gas. Cane massecuites that have not been heated above 156° F. do not usually foam. A crystallizer should always have provision for escape of the gases or preferably should be uncovered except while discharging by air-pressure. The same conditions as those observed in beet sugar manufacture are probably the cause of foaming in cane massecuites. J. A. Ambler<sup>7</sup> states that the action of amino-acids on glucose forms CO<sub>2</sub>, melanoidins and other substances, the melanoidins promoting frothing by lowering the surface tension. Ambler suggests the need of further fundamental research on this problem and its bearing on sugar manufacture.

<sup>5</sup> *Archief*, 1912, 20, 181.

<sup>6</sup> *Zeit. Ver. Zuckerind.*, 64, 543.

<sup>7</sup> Before the American Chemical Society, Swampscott, Mass.; Abstract Facts About Sugar, Sept. 29, 1928.

## CHAPTER XII

### EVAPORATING AND JUICE HEATING

#### Discussion of Methods. Methods of Calculation

BY

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**95. General Considerations.**—At the low pressures used in sugar house work the tables based on Regnault's formula

$$H = 1091.7 + 0.305 (T - 32) \text{ B.t.u.,}$$

or

$$H = 805.4 + 0.305 T \text{ calories,}$$

are too inaccurate for use. Only tables such as Table A, page 127, should be used.

**96. Boiling Point of Sugar Solutions.**—It is usual to assume that the temperature at which any portion of a given mass of water will boil is that corresponding to the pressure on the surface of the entire liquid mass. In sugar solutions this assumption leads to serious inaccuracies as the temperature of boiling of a portion of a mass of sugar solution will depend upon its density, its viscosity and the absolute pressure on the surface of the portion considered. This pressure will be that on the surface of the entire mass increased by the pressure due to the depth of the portion considered below the liquid's surface.

The variation in the boiling-point of sugar solutions with density is indicated in Fluoreno's table, page 433. In general, the variation is as in the following table:

Degree Brix.....	20	30	40	50	60
Add { Degree Fahrenheit...	1.1	1.8	2.7	3.6	5.4
Degree Centigrade...	.6	1.0	1.5	2.0	3.0

The boiling point of sugar solutions is also influenced by their purity.

**97. Effect of Viscosity.**—In the raw juice we sometimes have gums, mucilages and other compounds which increase the viscosity of the fluid. As anything which impedes the escape of the steam bubble from the heating surface tends to produce a local rise of temperature, it is easy to see that gummy

sirups will have a higher temperature of boiling than pure sugar solutions. The difference is uncertain.

**98. Variation with Depth Below the Surface.**—At the low pressure used in sugar house work small differences in pressure produce far larger temperature differences than under high pressures. For example: What will be the temperature at which a small portion of sirup of 50.6° Brix will boil if it is under a head of 2 feet of solid (no bubbles) sirup in the third effect if the vacuum is 26 inches?

A head of 2 feet of sirup of specific gravity 1.236 (50.6°) will change the pressure of 2 pounds per square inch, corresponding to 26 inches of vacuum, to 3.4 pounds per square inch. At 26 inches vacuum, water boils at 125.5° F.; under 3.4 pounds per square inch, or 23.8 inches of vacuum, it boils at 141.8° F. Add for density 4° F. and we have a temperature of 145.8° F. required to produce boiling in the sugar solution, without considering at all the effects of viscosity.

As transfer of heat from the heating to the heated liquid depends upon difference of temperature, we see that in this case there is a loss of  $145.8 - 125 = 20.3^\circ \text{F.}$ , difference in temperature as compared to a similar condition in the case of water. We shall call these *lost degrees in temperature* and later will discuss their effect.

**99. Specific Heat of Sugar Solutions.**—The relation between the density of a sugar solution and its specific heat is expressed by the equation of a straight line and hence intermediate values to those below may be readily found:

Degree Brix of solution.....	10	20	30	40	50	60	70	80	90
Specific heat, $C$ ....	.93	.86	.79	.72	.65	.58	.51	.44	.37

Water has the highest specific heat of any ordinary liquid, hence the less water in a liquid mixture the less the specific heat.

**100. Heat of the Liquid.**—To raise  $W$  pounds of a sugar solution whose specific heat is  $C$ , from a mean temperature,  $t_1$ , to a mean temperature,  $t_2$ , requires

$$WC(t_2 - t_1) \text{ B.t.u.}$$

Thus, to raise 10 pounds of sirup at 60° Brix from a mean temperature, 160° F., to a mean temperature, 180° F., would require  $10 \times .58(180 - 160) = 116 \text{ B.t.u.}$

**101. To Heat Sugar Solutions and Evaporate Water.**—The temperature of the vapor rising from a boiling sugar solution will be the same as that from pure boiling water under the same pressure. When the superheated bubble reaches the surface of a boiling sirup it loses all excess heat and temperature in vaporizing its watery envelope.

To raise  $W_1$  pounds of juice from a mean temperature,  $t_1$ , to a mean tem-

perature,  $t_2$  and evaporate  $W_2$  pounds of water at a temperature,  $t_3$ , corresponding to the pressure at the surface, requires  $W_1C(t_2 - t_1) + W_2L_3$ , where  $L_3$  is the latent heat of water corresponding to  $t_3$ .

In cases where  $t_1$  is greater than  $t_2$  the quantity  $W_1C(t_2 - t_1)$  is negative and must be subtracted.

**102. Condensate.**—In condensing steam in evaporators in sugar work, it is usual to assume that the condensate is removed at its boiling point. If taken to another place of less pressure and lower boiling point it will tend to self-evaporation and will give up a number of thermal units equal to the loss in temperature in the condensate multiplied by its weight. Thus, if  $W$  pound of steam is condensed at  $t_1$ , it will give up  $WL_1$  thermal units in its own vessel and if the condensate is led to another vessel, where steam is condensing at a lower temperature,  $t_2$ , it will there give up  $W(t_1 - t_2)$  B.t.u.

**103. Amount of Liquid to be Heated.**—The amount of normal cane juice must be increased by the amount of maceration water, milk of lime, wash-waters, and the water used in diluting the defecator-bottoms (mud or cachaza). There is a removal of water by evaporation in the various tanks and with the filter-press cake. In careful experiments, it is usual to calculate the weight of the purified juice to be evaporated or heated from that of the raw juice, taking into consideration the dilution as indicated by the densities. The dilution of the juice varies with the milling methods and the manufacture; also, with the equipment of the factory. The total weight of liquid to be heated or concentrated usually ranges from about 90 to 110 per cent of the weight of the cane. The mean evaporation in the multiple effect in tropical factories approximates 78 per cent by weight.

**104. Heat Losses.**—Heat is not only lost from the heating vessel itself by radiation but is lost from the pipes conveying heated liquid. Steam condenses in steam-pipes even when idle. The loss of heat is not negligible when the heating or evaporating is carelessly conducted.

**105. Coefficient of Heat Transfer ( $K$ ).**—The unit of heat transfer, called the Specific Thermal Conductivity, is the number of thermal units transferred per hour from the steam through the heating surface to the sugar solution for each degree difference of temperature, Fahrenheit, between the steam and the solution for each square foot of heating surface.

The formulas for heat transfer are simple, but they all involve this unit which may have any value between 1500 B.t.u. and 50 B.t.u. per hour. Experience and judgment are required to select a proper value. Some of the variables effecting the value of this unit are:

- (1) The velocity of the steam past the heating surface;
- (2) The velocity of the sugar solution past the heating surface;
- (3) The presence of air or other incondensable gases;
- (4) The character and thickness of the deposits on either or both sides of the heating surfaces;
- (5) The surface pressure and the depth, density and composition of the sugar solution being boiled. In certain experiments the heating surfaces were thoroughly cleaned. In three hours of use their heat transfer efficiency



dropped 50 per cent, then remained constant for a number of hours and then slowly decreased.

(6) The temperature of the steam and the rate of boiling.

The transfer of heat is the transfer of vibration and anything deadening the vibration results in loss. Steam molecules which have struck the surface and lowered their vibration rate should be swept aside to allow the impact of new molecules. On the sugar solution side of the heating surface, steam bubbles must be moved or swept away from the heating surface, as steam is a poor heat conductor. There is no longer any doubt as to the existence and enormous importance of surface films of gases, where the heating fluid is a gas, or of a vapor having air-containing films, as in condensers or effects, which magnify incalculably the resistance to the transfer of heat through the metal plate.

High current velocity then is desirable on both sides of the heating surface. To secure high steam velocity the cross-section of the steam current at right angles to its path should decrease as rapidly as the volume of the steam is decreased by condensation.

Economy is increased by securing a positive fluid circulation in a predetermined path. In the ordinary standard effect there is no definite path of either the steam or the juice. The juice circulation is erratic. In some spots the transfer of heat is high, with a tendency to cause the juice to spout into the steam space, but the largest part of the heating surface is not used efficiently. In the steam heating space there are large volumes in which the incondensable gases accumulate and lower the efficiency by blanketing the heating surfaces. As the steam is eddying with cyclonic velocity these volumes are constantly changing position. Eddy currents indicate inefficiency.

When exhaust steam is used for heating purposes, it is liable to contain much oil from the engines. The presence of this oil and of scale, arising from the lime used in the defecation, impede the transfer of heat. When turbines are used there is no oil in the exhaust.

As the metal used to separate the heated and heating fluids is generally either copper or brass and quite thin, the coefficient of heat transfer is not affected appreciably by its thickness.

**106. Evaporation of the Juice and Crystallization of the Sugar.**—Sugar juices are evaporated to the saturation or crystallization point in two distinct stages. The first operation is practically continuous while the second involves a cycle of operations repeated several times daily. If a unit of 10 cubic feet of juice of about 12.5° Brix is sent to a series of vessels called a multiple effect, it will be evaporated therein to about 2 cubic feet. This thickened juice is called sirup and its density is about 53° Brix. It is pumped from the multiple effect and is stored in tanks, preparatory to the next operation. In the second stage our unit of two cubic feet of sirup will be drawn into a vacuum pan and evaporated to about 1 cubic foot of massecuite which will be about half sugar and half molasses. Besides evaporating water from the sirup to bring it to the saturation point, the operations in the pan include graining or forming the sugar crystals and the subsequent enlargement of the crystals to commercial

size. This operation is called boiling to grain. Efficiency of evaporation governs the design of the multiple effect, while vacuum pan design is more

dependent upon efficiently handling and discharging a thick viscous mass quickly.

### 107. Multiple Effects.

—In Louisiana the multiple effect usually consists of two or three vessels operated in series, in Cuba three or four are used ordinarily; while in Europe five and even six have been used. Properly operated, efficiency increases in so far as fuel costs are concerned. These vessels are simple steam heaters where the condensing of steam on one side of a tube affords the heat necessary to evaporate water from the juice on the other side. In horizontal tube types, Fig. 36, the steam passes through the tubes but in vertical types, Fig. 37, the juice circulates vertically through the tubes. Necessarily the temperature and pressure of the steam on the heating side are higher than those on the juice or heated side. The vapor arising from the boiling juice in the first effect or vessel passes through a pipe to the steam space of the second effect, Fig. 35, and will evaporate water from the juice if the pressure maintained on this

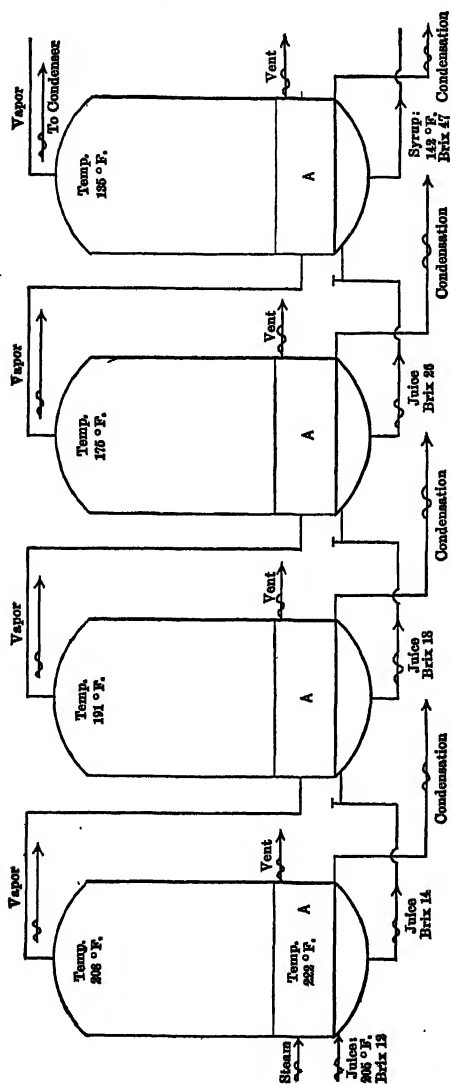


Fig. 35.—Diagram of Multiple-Effect Evaporator.

juice allows a proper fall of temperature between the two sides of the heating surface. The vapor arising in each effect does evaporative duty in the following one, except that from the last which is sent to a condenser.

In Fig. 35 the steam belt (A) could be of the form shown in section in Fig. 37. In this type the steam surrounds the small tubes. The juice boils up through the tubes and descends either through adjacent tubes or through the large down-take in the center. The large down-take may be omitted and several larger tubes used as down-takes as desired.

Note the following conditions in Fig. 35:

Effect.....	I	II	III	IV
Temperature differences.....	14°	17°	16°	40°
Pressures:				
Steam side.....	3.2 lb.	— 2.3 in.	—10.5 in.	—16.2 in.
Juice side.....	—2.3 in.	—10.5 in.	—16.2 in.	—24.7 in.
Differences.....	8.7 in.	8.2 in.	5.7 in.	8.5 in.
Densities, Brix				
Entering effects = 12°				
In effects.....	14°	18°	26°	47°

These data were obtained on test <sup>1</sup> and do not indicate either maximum capacity or maximum economy necessarily.

108. Disposal of Condensate.—The condensate in the steam belt of the first effect is free of sugar and may be used as boiler feed water. The condensate from the other effects, especially in the older types of evaporators, is liable to contain sugar carried over by entrainment in the rapidly moving steam from the preceding effect. There is rarely entrainment in modern well-proportioned and well-operated multiple effects. If the condensates containing even traces of sugar were used in the boilers, these would ultimately start foaming. In the modern factory, in which the condensates contain no sugar, this water is used in feeding the boilers, in saturating the bagasse, in diluting tank-bottoms (mud or cachaza) and in washing the various tanks. The condensate from the second effect may be passed into the third and the total into the fourth effect and then be removed by means of a pump. It is usual to pump the condensate separately from each calandria, as the heating section or belt of the effect is often termed.

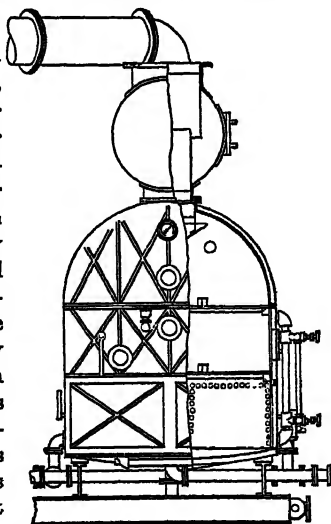


FIG. 36.—Horizontal Tube Type Evaporator.

<sup>1</sup> Figs. 35 to 38 are from a paper by E. W. Kerr in Transactions of the American Society of Mechanical Engineers, 1916.

**109. Disposal of Gases.**—Incondensable gases are brought into the multiple effect in the steam or juice and are given off under the reduced pressures and tend to accumulate, unless removed as fast as they enter. Certain nitrogenous bodies of the juice decompose and form ammonia during the heating. The path of the heating steam should be positive so that the gases are forced to move continuously to the exit where they are removed by the pipes which connect each calandria with the condenser, or air pump for the purpose. In the case of the vacuum pans using high pressure steam, the air is removed from the steam coils by valves in connection with the steam traps. The steam

traps let water pass, but neither air nor steam. The air relief valve will let the air escape but not the steam. Advantage is taken of the fact that air cools readily and will sink below the steam. After the air has escaped, the steam, in attempting to follow, heats a valve stem and so closes the valve that allowed air to escape.

**110. Juice Height.**—In the ordinary standard vertical multiple effect, Fig. 37, the greatest capacity is secured when the solid juice height, as indicated by the glass gage exterior to the effect, is one-third to one-fourth the tube height. Inside the effect the steam bubbles should just carry the juice over the tube sheet, but this does not indicate the juice level. The sirup is kept at the same height in all effects by hand-controlled valves or automatically, the amount pumped from the last effect being gaged by the amount of thin juice drawn into the first.

**111. Different Types of Multiple Effects.**—There are many different styles of multiple effects made of different modifications of the same few elements. The most common fault is too large juice capacity, so that the juice remains too long in the effect.

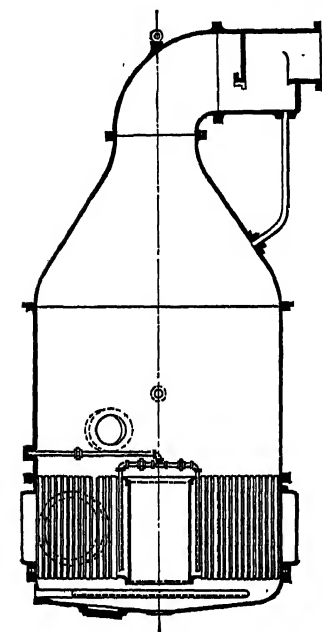


FIG. 37.—Vertical Tube Type Evaporator.

Some types lack in the positiveness of the circulation of the steam or juice or in the withdrawal of the incondensable gases.

The heating surface is made up of straight tubes usually either vertical or horizontal. Usually the steam passes through the horizontal tubes and the juice through the vertical ones. In one well-known type the juice is sprayed over inclined tubes containing steam. To permit air to escape the closed upper end of the tube contains a pin hole.

**112. Tubes.**—In the vertical standard effect the tubes are about  $\frac{1}{8}$  inch thick,  $1\frac{1}{2}$  inches or 2 inches in diameter and 40 to 54 inches long. The longer these tubes and the more intensely a small number of them is heated the more

certain will juice spout high up in the rapidly moving steam. The more efficient the heating surface, the more uniform will be the boiling over the entire top sheet and the smaller will be the temperature difference between the juice and steam sides. Intense localized heating should be avoided.

The tubes in horizontal effects are  $\frac{3}{4}$  inch or 1 inch in diameter and about 12 or 14 feet long. Longer tubes are liable to sway unless supported in the middle. These tubes are generally arranged in nests of eight, which may be removed as a unit (Fig. 36).

**113. Relations of Effects to Each Other.**—Multiple effects will operate when they are not operating efficiently. All the steam arising in one effect must be condensed in the next steam belt following, and conditions will change so that this will occur. Suppose that the third effect suddenly had its heating surface covered with scale. It could not transmit the amount of heat it transmitted before, so the steam pressure and temperature in its steam belt would rise till the heat transmitted was sufficient to condense all the steam now coming over from the second effect. But this would be less than formerly, as the rise in the steam pressure in the belt or calandria of the third effect has increased the pressure on the juice side of the second effect.

The capacity of each effect depends upon the capacity of the effect preceding it and on the one following.

**114. Real and Apparent Temperature Differences.**—The difference between the temperature in the steam calandria of the first effect and the temperature of the vapor going to the condenser from the last effect is the total *apparent* range of temperature. For a single effect, such as a vacuum pan, this total range occurs in one vessel. In the double effect this range is divided into two parts, in a triple effect into three parts and in a quadruple effect into four parts. For the causes enumerated in a preceding paragraph, there are lost degrees of temperature. These are tabulated below for a triple and quadruple effect, of the standard type for usual conditions under three heads.

(1) The boiling temperature loss is due to the fact that we are dealing with a sugar solution rather than with distilled water.

(2) The static head loss takes in the loss due to the depth of the boiling liquid.

(3) The third includes loss (assumed) due to the density and viscosity of the solution.

*To Obtain the Pressures to be Carried in the Vessels of the Multiple Effect.*—In the table below, the total loss degrees of temperature are  $5.4^{\circ} + 9^{\circ} + 15.4^{\circ} = 29.8^{\circ}$  F. The steam is assumed to enter the first vessel at  $227.2^{\circ}$  F. and the vapor to leave the last vessel at  $125.4^{\circ}$  F. The apparent range is  $227.2^{\circ} - 125.4^{\circ}$  and the real range is  $227.2^{\circ} - 125.4^{\circ} - 29.8^{\circ} = 72^{\circ}$  F. This real range can now be divided, for a triple effect, into three parts in accordance with conditions affecting the heat coefficients that have been considered, as for instance expected scale or oil on the heating surfaces. In the table the ranges of  $22^{\circ}$ ,  $24^{\circ}$  and  $26^{\circ}$  have been assumed. The apparent range in each vessel is the sum of the assumed real range in it and its expected lost degrees of temperature. To obtain the temperature of the vapor rising in the first vessel, sub-

tract the apparent range in temperature, 27.4° F., from the assumed steam temperature in the first steam belt. The 199.8° F. so obtained is the steam temperature in the second belt. Subtracting 33° F. from 199.8° gives 166.8° F., the temperature of the vapor in the second vessel. From the steam tables (page 127) we can readily find the pressures corresponding to the temperatures.

DISTRIBUTION IN A TRIPLE EFFECT

5-lb. Pressure	26-in. Vacuum		
Bodies	1	2	3
Boiling temp. loss, F°.....	0.9	1.5	5.4
Static head loss, F°.....	1.5	2	3
Viscosity loss, F°.....	3	5.5	7
Total losses, F°.....	5.4	9	15.4
Assumed effective temp. dif., F°.....	22	24	26
Apparent temp. dif., F°.....	27.4	33	41.4
Temperatures F°, 227.2.....	199.8	166.8	125.4
Pressure and vacua..... +5 lb.	-6.4 in.	-18.5 in.	-26 in.

DISTRIBUTION IN A QUADRUPLÉ EFFECT

Bodies	1	2	3	4
Boiling temp. loss.....	0.9	1	1.5	5.4
Static head loss.....	1.5	1.7	2	3
Viscosity loss.....	2.1	4.5	5.5	6.9
Total loss.....	4.5	7.2	9	15.3
Effective temp. dif., F°.....	16	16	18	18
Apparent temp. dif., F°.....	20.5	23.2	27	33.3
Temperatures, F°, 227.2.....	206.7	183.5	156	125.2
Pressure and vacua... +5 lb.	-3.4 in.	-13.4 in.	-21.2 in.	-26 in.

In "Steam Economy in the Sugar Factory," Abraham, translated by Bayle, the following table is given, illustrating losses in temperature in evaporators of beet sugar factories:

## LOSS IN TEMPERATURE, DIFFERENCE IN DEGREES FAHRENHEIT

Evaporation System	Individual Bodies					
	I	II	III	IV	V	Total
Single effect.....	15.3	....	....	....	....	15.3
Double effect.....	7.2	15.3	....	....	....	22.5
Triple effect.....	5.4	9	15.3	....	....	29.7
Quadruple effect.....	4.5	7.2	9	15.3	....	36
Quintuple effect.....	4.5	5.4	8.1	10.8	15.3	44.1

Hausbrand <sup>2</sup> gives even greater differences in the fall of temperature in the effects when the heating surfaces are of equal area. The ratios of the fall of temperature given by him are as follows:

In the double effect.....	1	1.58		
In the triple effect.....	1	1.44	3.44	
In the quadruple effect.....	1	1.105	1.48	2.175

Applying these ratios in the preceding cases would give apparent temperature differences as follows:

	°F.	°F.	°F.	°F.
In the triple effect.....	17.3	24.9	59	
In the quadruple effect.....	17.7	19.5	26.2	38.4

The discussion of the difference between real and apparent differences of temperatures has been introduced largely to show why the heat transfer coefficient varies in the effects when apparent temperature differences are used.

**115. Transfer of Heat.**—The number of B.t.u. transferred from the heating to the heated side per hour is  $KA(t_2 - t_1)$  where  $K$  = the mean thermal coefficient of conductivity per hour for the case considered;  $A$  = the area in square feet;  $(t_2 - t_1)$  = the difference of the mean temperatures of the steam and juice. The value of  $K$  depends upon whether  $(t_2 - t_1)$  is the difference of the apparent mean of real mean temperatures.

Tables B and C, pages 128, 131, taken from a paper on "The Capacity and Economy of Multiple Evaporators," by E. W. Kerr in the *Transactions of the American Society of Mechanical Engineers*, 1916, contain much useful information. Thus in experiment No. 27, Table B, the total range of temperature is  $14.5(1 + 1.14 + 1.10 + 2.76) = 87^\circ \text{ F}$ . The heat transfer coefficients are for the apparent temperature difference in the effects; the average is

$$(464 \times 1) + (408 \times 1.14) + (448 \times 1.10) + (193 \times 2.76) \div (1 + 1.14 + 1.10 + 2.76) = 325 \text{ B.t.u. Table B, Part III.}$$

<sup>2</sup> "Evaporating, Condensing and Cooling Apparatus," E. Hausbrand, Eng. Ed., 113.

The radical difference in the coefficient of heat transmission in the first three effects and the last one is due in part only to the greater number of lost degrees of temperature in the last effect. It is interesting to examine the coefficients given in Table 248. For equal cleanliness of surface and equally good circulation the coefficients should decrease in increasing amounts from the first to the fourth effect.

In Experiment 27, 837,836 pounds of water were evaporated in 7.13 hours with a total temperature range of 87° F., and a mean apparent coefficient of heat transfer of 325 B.t.u. Assuming roughly that it takes 970 B.t.u. to evaporate 1 pound of water, we find, using the total range of temperature, 87°, the area of the heating surface, *A*, in a single effect or in each effect of a multiple effect from the equation

$$325 \times 87 \times A = 970 \times 837,836 \div 7.13,$$

$$A = 4000 \text{ sq. ft., approximately.}$$

Care must be taken to evaluate all parts of the table in any test. Thus, test No. 35 gives 5.3 pounds of water evaporated per hour per square foot of heating surface. But the steam pressure in the first steam belt was 8.7 pounds below the atmospheric pressure. Evidently the heat in this test was transferred so readily that the steam pressure did not build up and if enough steam had been supplied to bring the pressure up to 18 pounds per square inch, absolute, the evaporation rate per

square foot would have been enormously increased. In this test the total range of temperature was 53.8° F., that in the first effect being 7.6° F.

The original papers by Prof. Kerr should be consulted before drawing conclusions as to the relative efficiency of the evaporators included in his tests. The results of his tests are given in the tables at the end of this chapter, to serve as examples.

**116. Heat Balance.** — In properly operated clean effects it is close enough for illustration but not for design to say that 1 pound weight of steam per unit

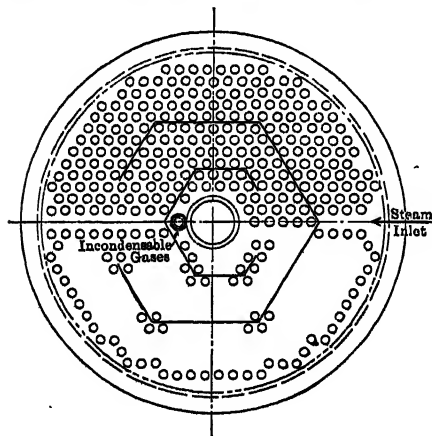


FIG. 38.—Vapor Distribution in Multiple Effect.

of time will evaporate approximately 3 pounds of water from 4 pounds of juice in a triple effect and that it will evaporate 4 pounds of water from 5 pounds of juice in a quadruple effect in the same unit of time. To show this, let us assume the steam temperatures on page 110 for a triple effect and also that the density of the entering juice is 12° Brix, the temperature being 180° F.



## HEAT DISTRIBUTION IN A TRIPLE EFFECT

First Effect	Heat	Juice, Pounds
1 lb. steam, 5 lbs. pressure per square inch. Latent = 960.7 B.t.u.		4.000
Heat required to raise 4 lbs. juice at 180° F. to 199.8° F., specific heat = 0.91		
$= (199.8 - 180) \times 4 \times 0.91 = \dots\dots\dots$	<u>72</u>	
Total heat liberated. . . . .	888.7	
Latent heat at 199.8° = 977.7 B.t.u.		
Evaporation No. 1 = $888.7 \div 977.7 = \dots\dots\dots$		.909
Juice transferred to No. 2 effect. . . . .		<u>3.091</u>

$$\frac{12}{\text{Brix}} = \frac{3.091}{4.00} \therefore \text{Brix} = 15.5^\circ.$$

## Second Effect

Heat liberated as follows:

Vapor from No. 1 effect. . . . .	888.7
Sensible heat from condensate:	
$(227.2 - 199.8) \times 1 = \dots\dots\dots$	27.4
Sensible heat from juice:	
$(199.8 - 166.8) 3.091 \times 0.85 = \dots\dots\dots$	<u>86.7</u>
Total heat liberated. . . . .	1002.8
Juice transferred to No. 2 effect. . . . .	3.091
Latent heat at 166.8° = 998	
Evaporation No. 2 = $1002.8 \div 998 = \dots\dots\dots$	1.005
Juice transferred to No. 3 effect. . . . .	<u>2.086</u>

$$\frac{15.5}{\text{Brix}} = \frac{2.086}{3.091} \therefore \text{Brix} = 23^\circ.$$

## Third Effect

Vapor from No. 2 effect. . . . .	1002.8
Sensible heat from condensate:	
$(199.8 - 166.8) (0.909 + 1.005) = \dots\dots\dots$	63.2
Sensible heat from the juice:	
$(166.8 - 125.4) (2.086) (0.75) = \dots\dots\dots$	<u>64.7</u>
Total heat liberated. . . . .	1130.7
Latent heat at 125° F. = 1021	
Evaporation in No. 3 = $\frac{1130.7}{1021} = \dots\dots\dots$	<u>1.107</u>
	0.979
Sirup from No. 3, $\frac{23}{\text{Brix}} = \frac{0.979}{2.086} \therefore \text{Brix} = 49.$	

**117. Increasing Evaporative Efficiency.**—Theoretically it is always advisable on the score of steam economy to use low-pressure steam to heat low-temperature solutions and high-temperature steam to heat those of high temperature. By using a series of heaters taking steam at different temperatures, solutions can be raised to high temperatures in a series of operations.

Thus, all the heat in the steam going to the condenser from the last effect is wasted, yet a portion of that heat could be used to raise cold juices to within some ten degrees of its own temperature. In other heaters, steam taken from the vapor pipes of the third, second, or first effect could raise the juice to higher temperatures.

The practical objections to the scheme are the increased complexity of the apparatus, its initial cost, upkeep and cost of cleanliness in avoiding bacterial action.

Let us make the following assumptions:

(1) The number of heat units in 1 pound weight of steam is the same at all pressures (practically).

(2) If  $x$  pounds of water are evaporated from a multiple effect, then the number of pounds of exhaust steam required will be  $x/n$ , if  $n$  is the number of vessels in the effect.

(3) If  $h$  pounds of steam are taken from the boilers and used in any single effect heater, such as the vacuum pan or defecator, the steam used will be  $\frac{x}{n} + h$ .

Suppose that the steam being sent from the last effect to the condenser were of high enough grade, it is evident that we could save 100 per cent of  $h$  pounds of steam by diverting  $h$  pounds of this steam from the condenser.

GAIN IN PER CENT BY ROBBING EFFECTS

Evaporating System	Steam Taken from Effect No. —				
	1	2	3	4	5
Single.....	100				
Double.....	50	100			
Triple.....	33 $\frac{1}{3}$	66 $\frac{2}{3}$	100		
Quadruple.....	25	50	75	100	
Quintuple.....	20	40	60	80	100

**118. General Equations.**—If we are to abstract  $h$  pounds of steam from only the first of  $n$  vessels and must evaporate  $x$  pounds of water in those vessels, we must furnish the first vessel with

$$(a) \quad \left( \frac{x}{n} - \frac{h_1}{n} \right) + h_1 \text{ pounds of steam.}$$

This expression may be considered to consist of two factors. We may imagine a weight of steam  $\left(\frac{x}{n} - \frac{h_1}{n}\right)$  to act normally in  $n$  vessels and so evaporate  $n\left(\frac{x}{n} - \frac{h_1}{n}\right) = x - h_1$  pounds of water. The other factor,  $h_1$ , evaporated the necessary  $h_1$  pounds of water from the first effect and this vapor is sent to perform the auxiliary heating.

If  $h_2$  pounds of steam are taken from only the second of  $n$  vessels, the amount of steam that must be sent to the first vessel will be

$$(b) \quad \left(\frac{x}{n} - \frac{2h_2}{n}\right) + h_2.$$

As before, we may imagine the steam represented by  $\left(\frac{x}{n} - \frac{2h_2}{n}\right)$  to act normally and supply  $n$  times its value in evaporation, or  $(x - 2h_2)$  pounds. The additional  $h_2$  pounds of steam will evaporate  $h_2$  pounds from *each* of two effects, giving  $x$  for the total evaporation and  $h_2$  for auxiliary heating.

In general let  $h_1, h_2, h_3, \dots, h_n$  pounds of steam be drawn from the first, second, third,  $\dots$   $n$ th effect, then the total consumption of steam will be, on rearranging expressions (a) and (b) above

$$\frac{x}{n} + h_1 + h_2 + h_3 + \dots + h_n - \frac{(h_1 + 2h_2 + 3h_3 + \dots + nh_n)}{n}.$$

**119. Saving in Per Cent.**—The saving  $h$ , may also be expressed as a percentage of  $x$ , so that the consumption of steam and the saving may be expressed in terms of  $x$ .

Thus, if  $h_1 \div x = p_1, h_2 \div x = p_2 \dots h_n \div x = p_n$ , the consumption of steam expressed as a percentage of that required for direct multiple effect evaporation would be

$$\frac{100}{n} + p_1 + p_2 \dots p_n - \frac{(p_1 + 2p_2 + 3p_3 \dots np_n)}{n},$$

and the percentage saving would be

$$(p_1 + 2p_2 + 3p_3 \dots np_n) \div n.$$

In a quadruple effect the first effect is robbed of .15 $x$ , the second effect of .10 $x$  and the third of .05 $x$ , the saving is

$$(.15 \times .25 + .10 \times .50 + .05 \times .75)x = \frac{12.5}{100}x \text{ or } 12.5 \text{ per cent.}$$

or

$$(.15 + 2 \times .10 + 3 \times .05) \div 4 = 12.5 \text{ per cent.}$$

In a quadruple effect if, in one instance, the first, second and third effects are robbed of equal amounts of steam and, in another instance the second

effect is alone robbed of the same quantity of steam, the efficiency is the same in both cases theoretically:

$$\frac{.25 + .50 + .75}{3} = .50.$$

Practically, however, the reduction in complexity of piping makes the robbing of the second effect preferable.

**120. Pauly Heaters or Pre-evaporators.**—When the supply of exhaust steam from the various engines is not sufficient there is a choice between admitting live steam direct from the boilers into the steam belt of the first effect or of using this live steam in the steam belt of a vessel called a Pauly, named for its inventor. The vapors from the Pauly may go either to the exhaust main or to the steam belt of the first effect. By connecting the steam main to the exhaust main by a reducing valve it is evident that when the exhaust pressure is low there will be a large demand made for live steam. When the exhaust pressure is high there will be little or no demand for live steam. The Pauly acts similar to a reducing valve, supplying the first effect with steam when the exhaust steam pressure falls.

The Pauly is of doubtful utility and its use is diminishing.

**121. Steam Consumption in a Cane Sugar House.**—In the following example the weight of steam used for any purpose is expressed as a percentage of the weight of the cane ground. It is further assumed that there is no difference in the quantity of heat in a pound of high-pressure steam and in that of a pound of low-pressure steam; that a pound of steam in condensing will evaporate a pound of water from the juice; that a preheater may be used and that a vacuum pan using low-pressure steam for heating is feasible.

In a house provided with a quadruple effect, the evaporation is 78 per cent of the cane weight and the exhaust steam supplied the first effect is therefore 78/4 per cent. The vacuum pans use 22 per cent and for heating 16 per cent steam is used. The weight of steam used is then, for,

(1) Straight evaporation, no preheater, no robbing of effects,

$$\frac{78}{4} + 16 + 22 = 57.5 \text{ per cent.}$$

(2) If a preheater receiving 10 per cent steam is used,

$$\frac{(78 - 10)}{4} + 16 + 22 = 55 \text{ per cent.}$$

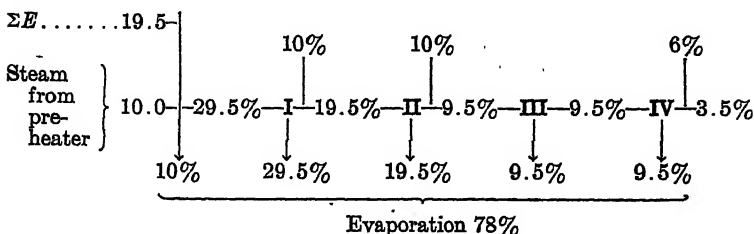
(3) If a preheater receiving 10 per cent steam is used and the first effect is robbed of 10 per cent steam for a low-pressure vacuum pan, the second effect is robbed of 10 per cent for juice heating and the fourth effect is robbed of 6 per cent for heating purposes,

$$\frac{(78 - 10)}{4} + 10 + 6 + 22 = \frac{10 \times 1 + 10 \times 2 + 6 \times 4}{4} = 41.5 \text{ per cent.}$$

If the effects are not robbed it is usual to give each vessel the same heating surface. This cannot be done if the effects are to be robbed as the mass of steam sent from one effect to the next diminishes greatly. Thus,

The exhaust steam sent to the first effect =  $\Sigma E =$

$$\frac{78}{4} - \frac{5 \times 10}{4} + 10 + 10 + 6 - \left( \frac{10 \times 1 + 10 \times 2 + 6 \times 4}{4} \right) = 19.5 \text{ per cent.}$$



Where a factory is prosperous, it is usual to increase its capacity. If changes are to be made in the sizes of effects, it is well to consider "robbing," letting the old effects follow a new first effect, enlarged to provide the necessary extra heating surface.

122. Heating Surfaces.—By referring to Table B, page 128, one is struck by the wide disparity in temperature fall allowed in the different effects in different houses, and also by the disparity in the coefficients of thermal conductivity in the different effects in houses of the same capacity.

It is evident that the results obtained when the effects are new and free of oil and incrustation, and are operated with the proper fall of temperature in each vessel, will be different when these economic conditions do not obtain.

Under average conditions, for example, we may assume that a quadruple effect will evaporate 6.5 to 7.5 pounds of water per square foot of heating surface per hour and hence we can obtain the heating surface necessary by dividing the water to be evaporated in each effect per hour by 6.5 to 7.5.<sup>3</sup>

Thus, if a quadruple effect is to evaporate 100,000 pounds of water per hour, the heating surface of each effect will be

$$100,000 \div (6.5 \times 4) = 4000 \text{ sq. ft., approximately.}$$

If the effects are robbed of vapor, the area of the heating surface of any effect is found by multiplying the weight of cane ground per hour by the product of the percentage of evaporation in that effect and the latent heat of the steam and dividing by the product of the apparent fall of temperature in that effect and its assumed coefficient of heat transmission in thermal units per hour. In the case of the first effect, the numerator of the fraction indicated should be increased by the quantity of heat in thermal units required to heat the juice from the temperature at which it enters the first effect to the temperature of boiling in that effect. The latent heat may often be assumed at 1000 thermal units for convenience.

<sup>3</sup> Under good conditions an evaporation of over 9 pounds has been reported.

The amount of evaporation in the first is to that in the fourth effect as 29.5 is to 9.5. (See diagram, page 117.) The steam sent to the condenser is only 3.5 per cent, thus reducing the quantity of injection water and air-pump capacity required.

Thus, suppose in case (3) above that the weight of juice = the weight of the cane (owing to the dilution by maceration water) is 125,000 pounds per hour; that it enters the effect at a temperature of 200° F. and is boiled at 213° F. Let the apparent temperature falls in a quadruple effect be 15° F., 17° F., 20° F., and 40° F., the corresponding coefficients of heat transmission be 450 B.t.u., 420 B.t.u., 400 B.t.u., and 200 B.t.u., and the latent heat be equal to 1000 B.t.u., approximately:

Heating surface of first effect

$$= \frac{125,000(0.9(213 - 200) + 0.295 \times 1000)}{15 \times 450} = 5680 \text{ sq. ft.}$$

Heating surface of second effect

$$= \frac{125,000(0.195 \times 1000)}{17 \times 420} = 3414 \text{ sq. ft.}$$

Heating surface of third effect

$$= \frac{125,000(0.095 \times 1000)}{20 \times 400} = 1484 \text{ sq. ft.}$$

Heating surface of fourth effect

$$= \frac{125,000(0.095 \times 1000)}{40 \times 200} = 1484 \text{ sq. ft.}$$

It is somewhat cheaper to make the effects of the same dimensions. This is also advisable under the usual operating conditions, in which the heating surfaces of the vessels containing the concentrated juice are more or less coated with scale. Evidently the dimensions of the third and fourth effects may be increased by diminishing the allowed fall of temperature in them and those in the first two decreased by increasing correspondingly the fall of temperature in them.

**EXAMPLE:** Find the area of the heating surface of a juice heater for a factory of 1000 tons of cane, daily grinding capacity; steam equal to 6 per cent of the weight of the cane to be taken from the vapor pipes of the second effect at a temperature of 183.5° F., the weight of the juice is that of the cane and its temperature entering the heater is 100° F., assumed coefficient of heat transfer is 250 B.t.u. per degree Fahrenheit difference between the two sides. Assume

the steam to lose 1000 B.t.u. per pound and that the specific heat of the juice is 0.9. The rise in temperature in the juice

$$= \frac{1000 \times 2000 \times 0.06 \times 1000 \text{ B.t.u.}}{1000 \times 2000 \times 0.9} = 66.6^\circ \text{ F.}$$

The mean difference of temperature between juice and steam sides of the heater

$$= 133.5 - \left( \frac{100 + 100 + 66.6}{2} \right) = 50.2^\circ \text{ F.}$$

Allowing 10 per cent for radiation and other losses, the heating surface will be

$$1.1 \times \frac{1000 \times 2000 \times 0.9 \times 66.6}{250 \times 24 \times 50.2} = 438 \text{ sq. ft.}$$

The original juice temperature plus its rise in temperature must be ten or more degrees below the assumed steam temperature.

**123. Heat from Bagasse.**—In burning wet bagasse there is more heat developed than is necessary to bring its moisture content to the boiling point, evaporate it, and superheat the vapor to 500° F. or over. If we assume that 1 pound of dry fiber in burning gives out 8300 B.t.u. then the heat content per pound of wet bagasse for various percentages of moisture content may be found from the table below:

HEAT CONTENT IN ONE POUND OF WET BAGASSE (CUBA)

Moisture content, per cent.....	42	44	46	48	50
Dry fiber, per cent.....	58	56	54	52	50
Heat of combustion.....	4814	4648	4482	4316	4150
To heat and superheat.....	511	535	559	583	607
Heat available.....	4303	4113	3923	3733	3543
Equivalent evaporation from and at 212° F.....	4.44	4.24	4.04	3.85	3.65
Equivalent steam:					
70 per cent boiler efficiency.....	3.10	2.97	2.83	2.70	2.55
65 per cent boiler efficiency.....	2.88	2.76	2.63	2.50	2.37
60 per cent boiler efficiency.....	2.60	2.54	2.43	2.31	2.19

Thus 1 pound of bagasse, containing 0.56 pound fiber would evaporate 2.76 pounds of water in a boiler of 65 per cent efficiency.

Assuming 10 per cent fiber content our unit of 100,000 pounds cane per day would give 10,000 pounds dry fiber. In addition there would be an extra 1000 pounds of glucose, sucrose and some nitrogenous compounds in the bagasse.

To get the weight of wet bagasse having the total dry fiber and the per cent of dry fiber, divide total dry fiber by the per cent of dry fiber content. Thus, dividing 11,000 pounds by an assumed dry fiber content 0.52, gives 21,139 pounds as the weight of wet bagasse as fired.

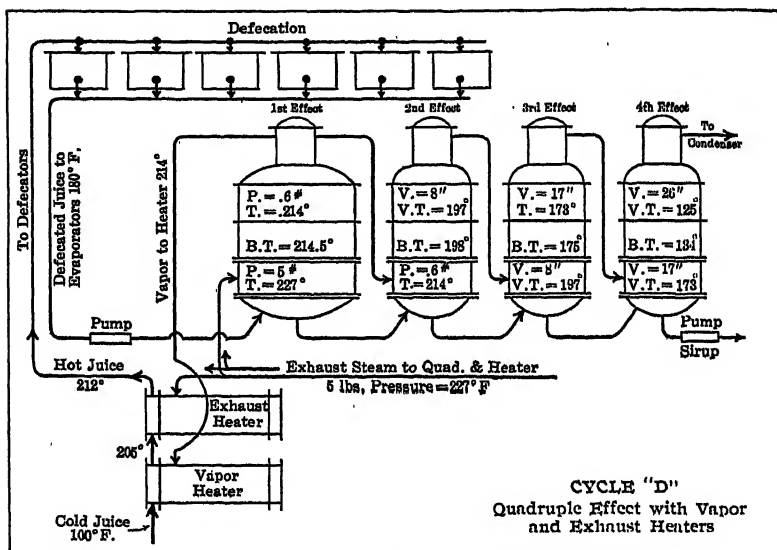


FIG. 39.—Quadruple with Vapor and Exhaust Heaters.

If we assume that 1 pound of wet bagasse in burning will evaporate 2.5 pounds water from and at 212° F., then the heat contained in 100,000 pounds cane would evaporate 52,825 pounds of water from and at 212° F.

**124. Power.**—In a plant grinding 100,000 pounds of cane per hour the requisite power will be about 1500 hp. Its equivalent in terms of the evaporation of water from and at 212° F. will be

$$\frac{1500 \times 33,000}{778 \times 970} \times 60 = 3935 \text{ pounds per hour.}$$

**125. Vacuum Pan and Radiation Losses.**—These are nearly constant losses and expressing these for our unit in terms of the evaporation of water



from and at 212° F. gives

	Water from and at 212° F.
Vacuum pans.....	20,000
Radiation losses.....	2,500

### 126. Evaporation in Multiple Effects following Webre and Robinson.—

Let us follow the scheme laid out in Fig. 39. It will be noted that both the first effect and an exhaust heater receive exhaust steam at 227° F. and that a vapor heater receives steam from the exhaust of the first effect. The cold juice is heated from 100° F. to 205° F. in the vapor heater and thence to 212° F. in the exhaust heater. The hot juice is sent to the defecators and thence goes to the first effect.

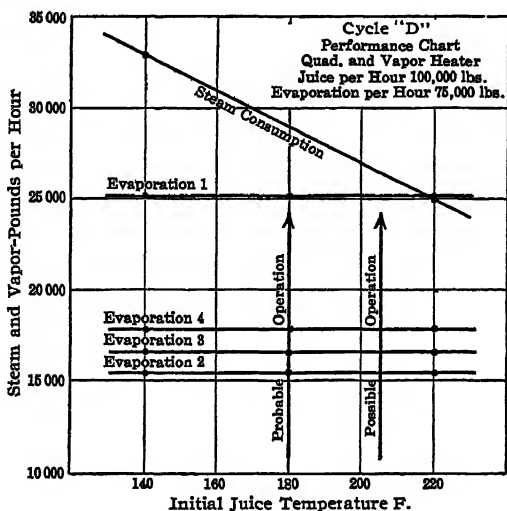


FIG. 40.—Performance Chart.

How many pounds of steam at 212° F. are required to reduce 100,000 pounds of juice to 25,000 pounds of sirup in a quadruple effect following the scheme in Fig. 39.

It is necessary to guess the required quantity, go through all the calculations given below and if the resultant weight of sirup differs materially from that required, namely 25,000, it will be necessary to make new assumptions and make new calculations until an assumption is made that gives the required sirup weight.

The temperature of the feed juice is very important. The steam consumption should be worked out for three different temperatures of the juice, and the results plotted as in Fig. 40. The heat balance shows that with the juice feed at 200° F. with this cycle the bagasse should furnish all the fuel that the house needs. (See Fig. 41.)

## QUADRUPLÉ WITH VAPOR HEATING CYCLE "D"

Bodies	Specifications	Heat, B.t.u.'s	Juice, Pounds
(1)	29,000 lb. at 227° = 29,000 lb. $\times$ 960.7 B.t.u./lb. =	27,850,000	100,000
	Deduct for heating 100,000 lb. $\times$ (214.5° - 180° = 34.5°) = .....	3,450,000	
	Available for Evaporation.....	24,400,000	
	L at 214° = 969.1 B.t.u./lb.		
	$E1 = \frac{24,400,000}{969.1} =$ .....		25,200
	Transferred to Number 2.....		74,800
(2)	Vapor ex. Number 1.....	24,400,000	
	Add Flash, 74,800 lb. $\times$ (214.5° - 198° = 16.5°) =	1,234,000	
	Available Total Heat.....	25,634,000	
	Deduct for Heater, 100,000 lb. $\times$ (205° - 100° = 105°) = .....	10,500,000	
	Available for Evaporation.....	15,134,000	
	L at 197° = 979.7 B.t.u./lb.		
	$E2 = \frac{15,134,000}{979.7} =$ .....		15,450
	Transferred to Number 3.....		59,350
(3)	Vapor ex. Number 2.....	15,134,000	
	Add Flash, 59,350 lb. $\times$ (198° - 175° = 23°) = ..	1,365,000	
	Available for Evaporation.....	16,499,000	
	L at 173° = 994 B.t.u./lb.		
	$E3 = \frac{16,499,000}{994} =$ .....		16,600
	Transferred to Number 4.....		42,750
(4)	Vapor ex. Number 3.....	16,499,000	
	Add Flash, 42,750 lb. $\times$ (175° - 134° = 41°) = ..	1,750,000	
	Available for Evaporation.....	18,249,000	
	L at 125° = 1021.6 B.t.u./lb.		
	$E4 = \frac{18,249,000}{1021.6} =$ .....		17,850
	Sirup ex. Number 4.....		24,900
	Steam from and at 212° = 29,000 lb. $\times$ .99 = 28,700 lb.		

In heating from 205° F. to 212° F. there was an expenditure of

$$\frac{100,000 \times 7}{970} = 720 \text{ units.}$$

The heat distribution then is

	Pounds water from and at 212° F.
Power.....	3,935
Heating from 205° to 212° F. (steam heat constant)...	720
Evaporation in quad and vapor heating.....	28,700
Vacuum pans.....	20,000
Radiation losses.....	2,500
Total.....	55,855
Available from bagasse alone.....	52,825*
Deficit.....	3,030

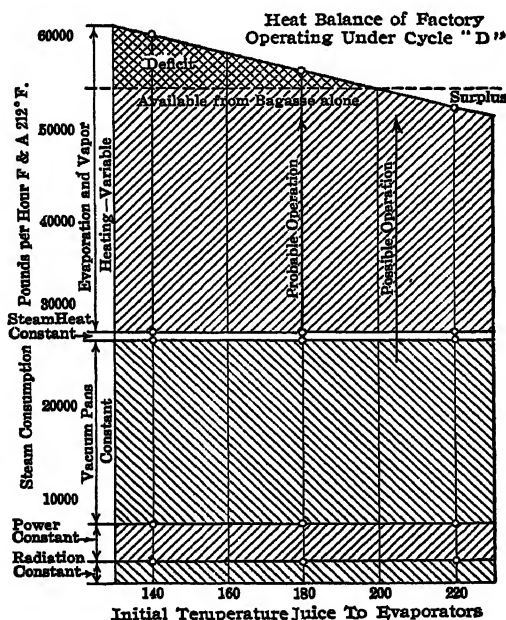


FIG. 41.—Factory Heat Balance.

\* See Steam Formation, Fig. 41.

**127. Vacuum Pan.**—A vacuum pan is a single effect, evaporating from 12 to 15 per cent of the quantity of water, required to be evaporated by the multiple effect. Economy of time rather than economy of steam is sought in its design. To boil and drop a strike quickly is desired. To boil quickly there are required properly clarified sirup, good sirup circulation, steam coils free of condensate and air and as great difference in temperature between the heating steam and the massecuite as is practicable. To drop a strike quickly the heating surfaces must not impede the free fall of the massecuite.

Boiler steam, passing a reducing valve, is admitted to a vertical manifold having 6 or 8 branches or nozzles entering the pan. Inside the pan these nozzles divide into 2 or 4 helical 4-inch copper tubes preferably not longer than 40 feet. In large pans there may be two manifolds on opposite sides of a diameter having similar nozzles and tubes. These tubes must be so arranged as to give the best circulation and the quickest drop possible.

No tube must contain steam unless it is covered with massecuite. In the first part of the operation the mass in the pan is rather thin and diminishes in volume till it is ready to form grain. After that period during the building up of grain the mass increases in density and amount to a maximum when the pan is full. Owing to lost degrees in temperature in boiling deep masses of massecuite, high-pressure steam, 45 pounds per square inch, is usually used. Sugar is often boiled with exhaust steam, particularly in calandria pans. It is common practice to boil coil pans with the lower coils on live steam and the top coils on exhaust. When the steam enters a tube at high velocity, as it does in the first part of the process, when the sirup is thin, and condenses, considerable unbalanced pressure results that sets up excessive vibration, unless the tube is restrained by collars fastened to rigid uprights. The soft coils must not rub against their collars and produce pin holes in the tube.

In designing the pan, about 0.55 boiler horsepower or 450 pounds of steam must be allowed per ton of cane per day.

**128. Condenser.**—By condensing the steam from the last effect or vacuum pan the pressure is reduced below that of the atmosphere. As the steam is accompanied by air and other incondensable gases, either dissolved in the original juices or sirup, or formed during the heating process or through leakage, an air pump must be provided to remove them so that the vacuum may be maintained. The barometric condenser is a preferred type on plantations. It is a condenser placed at a height above the ground so that it may discharge by gravity against the atmospheric pressure when the bottom of its discharge pipe is submerged in an open tank filled with water.

In the modern type the vapor pipe from the effect or pan enters the condenser at the side near the bottom and as the suction pipe of the air pump is attached at the top, the path of the gases is toward the top. The injection water enters near the top of the condenser and is broken up into sprays or thin sheets through which the vapors must pass on their way to the air pump opening. As the vapor and water move in opposite directions, it is sometimes called a counter-current condenser.

The discharge pipe usually supports the condenser. It should be about 34 feet high. To find the minimum height in feet, divide the difference

between the absolute pressure of the atmosphere and the minimum absolute pressure to be carried in the condenser in pounds per square inch, by the weight of a column of the discharge water 1 foot high and 1 square inch in cross-section. To this add the height of water in feet necessary to produce the assumed velocity of efflux of the discharge water.

Fins or projections should be placed on the bottom cone of the condenser proper to prevent the water from whirling and so rising above the normal level.

**Example.**—If 5 cubic feet of discharge water per second, weighing 60 pounds per cubic foot, are to be discharged from a discharge pipe of 0.5 square foot cross-section from a condenser carrying a pressure of 2 pounds, absolute, into the atmosphere at a pressure of 14.7 pounds per square inch, the minimum height will be found as follows:

$$(14.7 - 2) \div \frac{60}{144} = 30.5 \text{ feet;}$$

$$V^2 = 2gh; \therefore h = \frac{100}{64} = 1.56 \text{ feet;}$$

$$\text{Minimum height} = 30.5 + 1.56 = 32.06 \text{ feet.}$$

**129. Vacuum Pump.**—Cast iron is porous and air will leak through it, although it may be tight with regard to steam or sirup. A vacuum should be raised and the entire surfaces of effects and pans should be heated gently and then painted with a paraffine or other paint which will close these pores. Joints of all descriptions may leak air. The length of time a vessel will hold a vacuum indicates its air tightness. Air leakage may be two or more times the amount of air that must be handled unavoidably.

The office of the air pump is to pump air and other incondensable gases rather than vapor. The air and vapor come off as a mixture so that pumping some vapor is unavoidable, but this can be reduced to a very small amount if the air is cooled to a temperature not exceeding that of the incoming injection water by more than 10° or 15° F. To do this bring the vapor, just as it is entering the suction opening of the air pump, into contact with surfaces having the temperature of the incoming injection water.

The pressure in the condenser is nearly the same in all parts but the temperatures of the different parts of the vapor mass may differ considerably. If we take the temperature of the vapor and from the tables find the corresponding pressure we shall find this pressure less than that actually existing in the condenser. The difference of these pressures is the pressure due to the incondensable gases. The lower we cool the vapor the less the amount of steam in any given mass and the greater the amount of air as the total pressure remains constant. The proportion of vapor and of air present is in proportion to the pressures they exert. This is Dalton's law of mixed gases.

Thus suppose that the absolute pressure in a condenser as shown by the gauge was 102 mm., and that a thermometer shows that the temperature of the

vapors was 50° C. From the table we see that the pressure corresponding to this temperature is 92.3 mm., that is,  $\frac{P_2}{P_1} = \frac{92.3}{102}$ . The air pressure is 102 - 92.3 = 9.7 mm. At the top of the condenser let this vapor meet cooling surfaces at 15° C. and acquire a temperature of say 20° C. By the table we see that the steam pressure is now 17.5 mm., and, since the total pressure in the condenser is 102 mm., the air pressure is 102 - 17.5 = 84.5 mm. Each stroke of the air pump would remove  $\frac{84.5}{9.7} = 8.6$  times as much air as it would have removed under the previous conditions.

The work of the air pumps can be enormously reduced by

- (1) Reducing air leakage;
- (2) Using no more injection water than necessary;
- (3) Reducing the temperature of the vapors going to the air pump to a minimum;
- (4) Using pumps with no clearance volume.

**Example.**—Suppose 1000 kilograms of steam are sent to the condenser per hour and lose 600 calories (roughly) per kilo. Temperature of injection water is 15° C.; temperature of discharge water 43° C.; barometer 760 mm.; absolute pressure in the condenser is 102 mm.; temperature of vapor at orifice to air pump suction pipe is 20° C. As the amount of air in the sirup is small, it having been heated in clarification, and as the condensate from the first effect is used for boiler feed, we will assume in effects or vacuum pans that the injection water carries all the air into the condenser except air leakage. The latter will be assumed in this case equal to the amount of air carried in by the injection water. Assume that the volume of air at atmospheric pressure and at (273° + 15°) = 288° C., absolute, is 5 per cent of the volume of the water and that the efficiency of the air pump is 80 per cent. Find the volume of air pump displacement per minute:

One kilo steam requires 600 ÷ (43 - 15) = 21 kilos injection water.

Each kilo of injection water contains 0.05 liter air at 760 mm. and 288° C., absolute. At 84.5 mm. and 293° C. absolute, this becomes 0.46 liter by applying equations

$$\frac{P_1 V_1}{T_1} = \frac{P_2 V_2}{T_2} \quad \text{or} \quad \frac{760 \times 0.05}{288} = \frac{84.5}{293} V_2.$$

Displacement volume of air pump per minute is

$$\frac{(21 \times 1000 \times 0.46)2 \times 100}{60 \times 80} = 402 \text{ liters.}$$

The effect of increased leakage or higher temperature of gases passing into the condenser is readily calculated.

130.—TABLE A.—PROPERTIES OF SATURATED STEAM AT DIFFERENT VACUA AND PRESSURES

Vacuum	Temp., F.	Cubic Feet per Pound	Latent Heat, B.t.u.	Vacuum	Temp., F.	Cubic Feet per Pound	Latent Heat, B.t.u.
28.0 in.	101.15	339.6	1035	10 in.	192.23	39.21	983
27.5	108.70	275.2	1030	9	194.52	37.4	981
27.0	115.06	231.9	1027	8	196.73	35.8	980
26.5	120.55	200.2	1024	7	198.87	34.3	978
26.0	125.38	176.7	1021	6	200.94	33.0	977
25.5	129.75	158.1	1019	5	202.92	31.8	976
25	133.77	143.0	1017	4	204.85	30.6	975
24	140.64	129.0	1013	3	206.71	29.5	974
23	146.78	104.5	1009	2	208.52	28.6	973
22	152.16	92.3	1006	1	210.28	27.7	972
21	157.00	82.6	1003	0	212.00	26.8	970
20	161.42	74.8	1001	1 lb.	215.3	25.2	968
19	165.42	68.5	998	2	218.5	23.8	965
18	169.14	63.1	996	3	221.5	22.5	964
17	172.63	58.6	994	4	224.4	21.4	962
16	175.93	54.5	992	5	227.2	20.4	961
15	179.03	51.2	990	6	229.8	19.5	959
14	181.92	49.0	989	7	232.4	18.6	957
13	184.68	45.5	987	8	234.8	17.8	955
12	187.31	43.2	985	9	237.1	17.1	954
11	189.83	41.1	984	10	239.4	16.5	953

131.—TABLE B, PART I.—PRINCIPAL DATA FROM TESTS OF SUGAR HOUSE EVAPORATORS (E. W. KERR)

(Description of the Evaporators)

Number	Type (1)	Number of Bodies	Rated Capacity, Gallons per 24 Hours, 75 Per Cent Evaporation	Length of Tubes, Feet	Diameter of Tubes, Inches	Heating Surfaces of Each Body in Square Feet	Method of Removing Condensation. See Footnote *	Method of Venting* (See Footnotes)	Number of Days Since Cleaned
1	A	4	290,000	4.5	2	4504.8	<i>f</i>	<i>bc</i>	6
2	A	3	270,000	4	2	3740	.....	.....	3
3	A	3	290,000	4	2	3983	<i>e</i>	None	3
4	A	3	185,000	4	2	2579	<i>e</i>	<i>bd</i>	0
5	A	3	280,000	4	2	.....	<i>e</i>	Note 1	0
6	A	3	210,000	4	2	2855	<i>e</i>	None	7
14	B	4	145,000	9.5	$\frac{3}{4}$	2000	Note 2	<i>d</i>	6
15	B	4	145,000	9.5	$\frac{3}{4}$	2000	Note 2	<i>d</i>	3.5
16	B	4	145,000	9.5	$\frac{3}{4}$	2000	Note 2	<i>d</i>	7
17	B	4	250,000	13	$\frac{3}{4}$	3432	<i>e</i>	<i>c</i>	0
18	B	4	250,000	13	$\frac{3}{4}$	3432	<i>e</i>	<i>c</i>	0
23	C	4	400,000	7.25	4.5	2570	<i>g</i>	Spec.	0
24	C	4	400,000	7.25	4.5	2570	<i>g</i>	Spec.	0
25	C	4	400,000	7.25	4.5	2410	<i>g</i>	Spec.	0
27	D	4	400,000	.....	...	4000	<i>f</i>	Note 3	3
28	D	4	400,000	.....	...	4000	<i>f</i>	Note 3	2
29	D	4	400,000	.....	...	4000	<i>f</i>	Note 3	0
30	D	3	200,000	23	...	2112	<i>f</i>	None	0
33	E	3	220,000	4	2	3054	<i>f</i>	Note 4	1
34	E	3	.....	4	2	2683	<i>f</i>	Note 4	2
35	E	3	.....	4	2	2683	<i>f</i>	Note 4	7
38	G	4	290,000	4	2	1529	<i>e</i>	<i>h</i>	3

\* *a*, Through shell at side; *b*, through top tube sheet; *c*, direct to condenser; *d*, body to body; *e*, pumps each body; *f*, syphon body to body; *g*, barometric leg pipe; *h*, each tube vented.

Note 1. Vented through 12 holes  $\frac{1}{16}$  inch each, direct to vapor space.

Note 2. Centrifugal pumps each body.

Note 3. 2-inch vents at side, bottom, and top, body to body.

Note 4. See Fig. 92.

(1) A = "Standard," vertical submerged tubes; B = Jelinek; C = Lillie; D = Kestner; E = Webber's "Standard"; G = Sanborn.



132.—TABLE B, PART II.—PRINCIPAL DATA FROM TESTS OF SUGAR HOUSE EVAPORATORS (E. W. KERR)  
(Refer to Part I for Descriptions of Evaporators)

Number	Duration of Test, Hours	Type	Number of Bodies	Steam Pressure		Temperature, Deg. Fahr.		Density, Deg. Brix at 17.5° C.		Evaporation, Per Cent.		Juice Treated per 24 Hours, Equivalent to 75 Per Cent Evaporation, Per Cent Rated Capacity	Water Evaporated, Pounds	Water Evaporated per Pound of Steam Supplied, Pounds	Water Evaporated per Square Foot Heating Surface per Hour, Pounds	Thermal Efficiency, Per Cent
				First Body, Pounds, Abs.	Last Body, Inches, Abs.	Juice Entering	Juice Leaving	Juice Entering	Juice Leaving	By Weight	By Volume					
1	7.13	A	4	16.3	1.8	183.5	101	13.7	50.2	72.7	76.6	137	587,914	.....	5.44	85.06
2	6	A	4	14.6	3.6	185.9	134.2	15.2	55.5	72.6	77	.....	350,584	.....	5.19	89.05
3	6.66	A	3	15.3	3.2	188.6	.....	13.2	52.2	74.7	78.6	102	505,304	.....	6.35	.....
4	12	A	3	15.6	3.4	198.4	.....	15.4	61.1	74.7	79.4	138	933,684	.....	10.38	.....
5	5.75	A	3	16.7	4.9	183.8	.....	13.6	51	73.4	77.4	83	327,466	.....	4.96	.....
6	4.33	A	3	14.7	3.5	194	124.5	13.3	53.2	77.2	81.3	87	195,763	.....	5.27	.....
14	4	B	4	22.9	3.8	199.7	119.8	18.1	55.9	67.6	72.5	109	155,198	3.52	4.85	.....
15	4	B	4	21.4	4.0	199.7	129.2	17.5	62.9	72.2	77.2	174	248,701	3.7	7.77	85.06
16	6	B	4	23.4	4.0	200.5	127.3	16	47.2	66.1	70.3	107	225,586	.....	4.69	.....
17	6	B	4	18.7	3.7	181	.....	13	57.5	77.5	81.4	86	314,062	3.4	5.08	90.48
18	4.5	B	4	17.4	5	196.5	140	13	59.3	78.1	82.1	116	319,833	.....	5.18	.....
23	6	C	4	19.1	5.9	194	141	12.7	53.2	76.1	79.9	94	556,923	3.71	9.02	96.9
24	6	C	4	17.7	6.5	191.8	144	13.1	58.7	77.7	81.7	94	553,877	3.95	8.98	97.5
25	6.06	C	4	17.9	4.6	188.8	131	12.7	65.4	80.6	84.6	76	450,293	3.69	7.71	97.2
27	7.13	D	4	17.9	5.2	205	141.8	11.6	47.3	75.5	78.9	138	837,836	4.21	7.34	97.2
28	6.03	D	4	17.9	4.9	207.7	136.9	12.6	46.2	72.7	76.7	131	748,958	4.24	7.76	94.8
29	6	D	4	18.1	5.2	208.8	132.6	12.8	45.4	71.8	75.5	124	790,172	4.28	8.23	93.3
30	4	D	3	14.8	2.3	212.2	125	16.5	46.2	64.3	68.5	93	191,585	2.91	7.77	86.5
33	6	E	3	14.6	4.6	171	136	14.9	58	75.4	78.8	105	338,859	2.49	6.27	97.4
34	5.39	E	3	11.06	5	184.6	129.2	15	50.8	70.6	74.8	.....	190,517	.....	4.4	.....
35	4	E	3	6.07	3.2	179	.....	14.7	51	73	75.2	.....	156,176	.....	5.3	.....
38	5.69	G	4	15.4	5.8	188.1	147.4	15	64.2	76.7	81.2	.....	206,917	.....	5.95	.....

133.—TABLE B, PART III.—RELATIVE TEMPERATURE FALL AND COEFFICIENT OF HEAT TRANSMISSION  
(Data from Tests on Sugar House Evaporators, E. W. Kerr)

No.	Temp. Fall, 1st Body, Deg. Fahr.	Relative Temperature Fall				Coefficient of Heat Transmission				No. of Days Since Cleaned
		1st Body	2d Body	3d Body	4th Body	1st Body	2d Body	3d Body	4th Body	
1	17.4	1.0	0.67	0.87	4.41	315	430	372	62	172
2	14.8	1.0	1.89	3.21	.....	556	163	111	.....	200
3	33.4	1.0	0.27	1.62	.....	174	663	114	.....	184
4	17.3	1.0	1.22	3.31	.....	447	355	147	.....	247
5	14.0	1.0	1.85	3.08	.....	371	170	113	.....	174
6	4.4	1.0	8.30	11.61	.....	1185	126	107	.....	166
14	18.9	1.0	0.69	1.85	2.87	239	270	97	79	130
15	21.1	1.0	0.52	1.22	2.50	321	508	203	90	202
16	19.2	1.0	0.66	1.88	2.24	192	344	115	113	154
17	17.9	1.0	0.95	1.26	2.45	220	202	161	87	146
23	15.0	1.0	0.80	0.93	2.93	545	648	600	207	394
24	14.0	1.0	0.95	1.07	2.52	610	593	552	286	449
25	11.6	1.0	1.02	1.27	4.62	680	644	505	143	334
27	14.5	1.0	1.14	1.10	2.76	464	408	448	193	325
28	15.2	1.0	1.11	1.14	2.60	444	405	426	209	328
29	18.0	1.0	0.67	1.01	2.17	397	605	378	244	353
30	16.1	1.0	1.50	4.14	.....	402	287	107	.....	192
33	19.8	1.0	0.75	2.01	.....	317	393	152	.....	244
34	5.0	1.0	3.29	6.38	.....	769	219	130	.....	217
35	7.6	1.0	1.18	4.91	.....	623	558	149	.....	284
38	8.0	1.0	1.13	1.88	5.38	682	571	365	138	293

134.—TABLE C.—SUMMARY OF STEAM CONSUMPTION TESTS

	Condensation per Ton Cane	Steam <i>F</i> and <i>A</i> 212° F. per Ton of Cane	Equivalent Pounds Steam at Boiler per Ton Cane	Steam <i>F</i> and <i>A</i> 212° F. Per Cent of Cane	Steam <i>F</i> and <i>A</i> 212° F. Per Cent of Total	Boiler Horse-power per Ton of Cane	Boiler Horse-power 4500 Tons Cane per 24 Hours
	Pounds	Pounds					
1. Evaporators.....	417.0	413.7	410.0	20.67	28.2	.9485	2242.3
2. Vacuum pans, "straight" strikes..	191.4	192.7	191.0	9.63	13.17	.2323	1045.3
3. Vacuum pans, mixed strikes.....	164.2	164.87	163.4	8.24	11.02	.1887	849.2
4. Vacuum pans, crystallizer strikes..	89.32	90.0	89.2	4.5	6.11	.1084	477.8
5. Vacuum pans, average.....	444.92	447.57	443.6	22.37	30.3	.5395	2427.7
6. Live steam heaters.....	81.5	81.4	80.7	4.07	5.52	.0977	439.6
7. Exhaust steam heaters.....	210.7	208.6	206.7	10.43	14.16	.2512	1130.4
8. Kestner heater.....	28.0	27.75	27.5	1.39	1.88	.0332	149.2
9. Equivalent of mechanical work....	.....	54.66	54.17	2.73	3.72	.0657	295.65
10. Total accounted for in factory....	1182.12	1233.68	1222.67	61.67	83.7	1.4859	6086.5
11. Radiation and unaccounted for assumed.....	.....	239.0	236.8	12.00	16.22	.2790	1246
12. Total delivered by boilers †.....	.....	1479.68	1459.47	73.67	100.00	1.7649	7932.5

\* By Prof. E. W. Kerr, Guanica, P. R.

† Based upon the assumed radiation and unaccounted for of 12 per cent.

## CHAPTER XIII

### PURGING AND PACKING THE SUGAR

**135. Centrifugal Work. Curing and Packing.**—The sugar is purged of molasses by centrifugal force in a machine called a "centrifugal." This machine is essentially a perforated drum or basket so arranged that it may be revolved at high velocity. The basket revolves within an iron casing called the "curb" which serves to catch the molasses and lead it to the conduit. The basket is lined first with a brass wire "backing" screen of 10 meshes to the inch inside of which is a perforated sheet of brass or bronze known as the "lining." The older type of lining was perforated with round holes having from 400 to 625 perforations to the square inch, but the screen now generally used has slotted apertures  $.012 \times \frac{5}{8}$  inch tapered from the inside outward to more than twice the inside width in order to give a freer flow of the molasses. The slotted linings permit of the use of much heavier metal than the old round-hole type. The older centrifugal baskets were 30 inches in diameter and occasionally 36, but all modern factories use 40-inch centrifugals 24 inches deep and upward, which have a speed of about 1000 revolutions per minute.

The massecuite is fed into the machines from a mixer which prevents the crystals from settling. The centrifugal usually receives its charge of massecuite while it is in motion, though it may be charged at rest. The basket is spun until the sugar is practically free of molasses. The molasses passes through the perforations of the lining and the crystals are retained. In making high polarization sugars and a white product, as soon as the surface of the wall of sugar appears in the centrifugal, indicating that little molasses is left, it is washed with water or special washes. This treatment removes the film of molasses that adheres to the crystals. Special washes are occasionally used to color the crystals to meet certain market conditions, *e.g.*, the Demarara crystals.

Centrifugals are sometimes arranged with double gutters to classify the molasses. After washing begins the molasses is directed into the second gutter and is returned to the pan-room for reboiling with rich sirup. This arrangement is not entirely satisfactory on account of the lagging behind of the heavy molasses first thrown off, which in part mixes with the wash. The classification is better accomplished by double purging as described farther on.

Each sugar-drier or purger manipulates two and sometimes three centrifugals. A 40-inch by 24-inch centrifugal, driven by belts and working with one operative to two machines, should purge from 5000 to 6000 pounds of 96° polarization sugar per hour, the quantity varying with the skill of the operative, the quality of the massecuite and the facility with which the centrifugal may

be started and stopped. This capacity may be greatly increased by the use of mechanical dischargers, while self-discharging baskets will give double this capacity.

The discharger is a plow arrangement that is lowered into the basket, and directed against the wall of sugar. The basket is revolved slowly against the plow, which cuts down the sugar and pushes it out of the machine by the bottom valve. There are many makes of these, all of the same general design, and their use is common in cane, refinery and beet-house work.

The self-discharging basket has a steep conical section at the bottom and may or may not have a discharge-valve. The commoner type is without discharge-valve and has a deflector on the spindle or shaft to direct the massecuite toward the wall of the basket. In the other type, the valve may be raised while the centrifugal is running. The machine with a deflector is charged with massecuite while running. When the sugar has been purged and the centrifugal is stopped the sugar usually falls out of it without assistance. In the other form it is necessary to lift the valve before the centrifugal is stopped. These machines are used only with freely purging sugar of strong grain such as high-grade massecuites and refinery affination magmas. They are not used for refined sugars because of the danger of contaminating the purged sugar with drippings of massecuite.

The manufacture of white sugar demands special care in purging. The sugar is washed in the centrifugal with large quantities of water, from two to six gallons being required per charge in a 40-inch machine. A small quantity of ultramarine is sometimes added to the wash water to kill the yellow tinge of direct consumption sugars but is no longer used by bone-black refineries. The method of double purging gives the best results in washing plantation white sugars.

**136. Double Purging.**—Two sets of centrifugals are necessary for double purging. The sugar is purged in the first set without washing or with very little water. It is then "cut down" into a mingling device and formed into a magma with sirup from the second purging. The magma is elevated to the second mixer and is purged in the second set of centrifugals with thorough washing.

Double purging serves a two-fold purpose: It separates the dark, low-purity molasses of the first purging from the rich, light-colored sirup of the second. This sirup is of the suitable color and richness to permit reboiling with cane-sirup to make white sugar. The mingling process mixes the crystals thoroughly with the light-colored sirup. The friction of crystal against crystal promotes the removal of the adhering film of molasses.

Double purging was introduced into Cuba by Dr. Spencer to facilitate the handling of crystallizer-sugar in the production of one grade of sugar and final molasses. (89) The crystallizer-sugar is purged without washing and the molasses from it is final. The sugar is discharged into a mingling screw-conveyor and transferred to the mingler proper. The conveyor-screw is lubricated with first molasses or sirup diluted to about 78° Brix, and this molasses or sirup is also used in the mingler to form the magma. The mingler is simply a mixer. The magma is pumped to the first machine's mixer for immediate

purging or to a storage crystallizer to be used as a footing or "seed" in boiling massecuites.

**137. Packing.**—Raw sugars are usually packed in jute bags for shipment, and in Cuba, without further drying than they receive in the centrifugals. Their moisture may be reduced by the use of superheated steam in the centrifugal. Raw sugars may also be dried in granulators or driers such as are used in the refineries (see page 179) provided they are of high test and clean crystal. This method of drying is very liable to result in a product that will harden or cake in the packages.

The usual Cuban sugar bag holds from 325 to 330 pounds of centrifugal sugar, measures 29 by 48 or 30 by 50 inches and its tare is from 2.5 to 2.7 pounds. Porto Rico packs in bags holding either 250 or 310 pounds and the Hawaiian Islands use bags of 125 pounds capacity.

White sugar may be dried in the granulator or dryer or with superheated steam in the centrifugal. The crystals lose a part of their gloss in the granulator through friction with one another. The American market is accustomed to such sugar and possibly prefers it. The gloss is preserved in drying with superheated steam and it is such sugars that are made in Java for the home and East Indies markets. The steam should be heated to about 200° C. in a separate boiler.

Various types of conveyors are used in transferring sugar from the centrifugals to the packing-bins. Sugar should not fall directly from the centrifugal into the package, since under this condition it cannot be of uniform quality, and being moist and warm will tend to harden. Three types of conveyors are in general use: (1) Ribbon- or screw-conveyor. This is a screw or spiral ribbon that revolves in a trough and carries the sugar with it. The cross-section of the trough should be a parabola (2) Endless belt. The belt is often of curved-steel slats attached to a chain. This is a very efficient device, but has the objectionable feature of leakage of sugar. (3) Grasshopper conveyor. This is an efficient conveyor that is used very generally in factories of Scotch design. As is implied by the name, the motion of the conveyor is something like that of a grasshopper. A trough is arranged to move slowly forward, carrying the sugar, and then pull backward very quickly, leaving the sugar. Each stroke advances the sugar a certain distance. All parts of the conveyor are easily accessible for cleaning. The packing bins are generally large enough to permit some cooling of the sugar by keeping it in storage a short time.

In most modern plants the sugar is discharged into the bags from automatic scales connected with these storage bins. These scales have been perfected so that with careful supervision they deliver sufficiently exact weights for control purposes. (376.)

**138. Caking of Sugars.**—Caking or hardening of both raw and refined sugars is a frequent trouble, particularly when sugar has been exposed to a sharp drop in temperature. The generally accepted theory of the cause of caking is that there is a supersaturated film of sirup around the crystals at a given temperature, and with a fall in temperature, or a reduction in the moist-

ure content of the film, crystallization starts up, cementing the original crystals of sugar together with these newly-formed, fine crystals.<sup>1</sup>

In accord with this theory (and also with Cuban experience), are some conclusions from a study of the immediate causes of caking of raw sugars by Wuthrich:<sup>2</sup>

1. *Sugar of High Moisture Content*.—Sugar with a high moisture content, say 1 per cent and more, was found invariably to cake when brought into surroundings where part of its moisture evaporated or "dried out." On the other hand, it was found that caking did not occur as long as the moisture content remained high.

2. *Sugar of Low Moisture Content*, (0.3 to 0.4 Per Cent.).—Normal sugar with low moisture content will not cake, whether cold or hot bagged, as long as it is not exposed to a very humid atmosphere and later on to a dry atmosphere.

3. *Quality of the Sugars*.—The foregoing remarks refer to "normal sugars." For if the sugar be abnormally viscous and sticky, it will cake no matter whether it has a low- or high-moisture content; and no matter whether it is bagged cold or hot.

Further, small grain sugar is much more susceptible to caking than large grain sugar. Especially sugar with high-moisture content cakes very soon when the grain is small. The smaller the grain, the larger the surface of a given quantity of sugar, and the greater the chance of its being cemented together.

4. *Temperature at Bagging*.—Hot bagging of sugar of high moisture content will increase the chance of caking considerably, since it helps the water to evaporate. Hot bagging of sugar of low water content does not influence the caking of normal sugar, but does do so when the sugar is abnormally viscous and sticky.

5. *Weather and Climatic Conditions*.—From the above the conclusion may be drawn that weather conditions and change in these conditions, in both the country of origin and destination, and also a difference in the climatic condition of these countries, form a great factor in the formation of caked sugar.

6. *Storage Conditions*.—If the air in a warehouse becomes too hot or too moist, this will increase the chance of caking.

It is seen that raw sugars which are of good even grain, well dried, and not excessively gummy (good clarification) are not likely to cake. It has been shown elsewhere (p. 137) that these are the characteristics of a good keeping sugar (one that will not deteriorate in test). Packing raws cool and storing them in well-constructed, dry warehouses is also conducive to the prevention of caking. Nevertheless, practically all raw sugar will harden when exposed to freezing weather, softening again when the weather moderates.

The caking of well-made refined sugars is largely a matter of storage conditions. Sugar of irregular grain, particularly one containing an excess of dust, will tend to cake more easily while a sugar coming from the granulators with more than a trace (.03 — .05 per cent) of moisture will also cake in storage, but high-grade refined sugars are not generally subject to these criticisms. The usual cause of the difficulty is through the absorption of moisture from the surrounding atmosphere with a subsequent drying out or drop in

<sup>1</sup> H. S. Walker. *Int. Sug. Jour.*, 25 (1923), 131.

<sup>2</sup> *Int. Sug. Jour.*, 25 (1923), 192.

temperature, setting up the crystallization of fine grain in the moisture film. This effect of atmospheric conditions during storage is now so well recognized that many American refineries, located in sections where high humidity prevails, have lately installed warm-air ventilating systems in their refined warehouses, the idea being to keep the sugar in an atmosphere of lower "relative humidity" than the outdoor air, thus preventing moisture absorption by the stacks of sugar.

Wuthrich<sup>3</sup> gives an illustration of the effect of moisture absorption on high-test dry Java white sugars. Sugar from the same crystallizer was transported for fourteen days in a hot humid atmosphere, part in open trucks and part in closed. The sugar conveyed in open trucks caked badly, that in closed trucks not at all.

**139. Classification of Raw Sugars.**—The basis of the American market is usually a centrifugal sugar polarizing 96°. The corresponding sugar in Java is of higher test and is there called "muscovado," and in the American market "Javas." The next grade is 89° molasses sugar or seconds. This formerly was always a sugar crystallized at rest, but now low-testing grain-sugars are included. Muscovado sugar is that made by open-air evaporation and crystallization in coolers at rest. The molasses is removed by simple drainage. Such sugars are now rarely seen in the American market, the bulk of the raw product of the Tropics being of the 96° grade.

Raw sugars entering the United States were formerly classified according to the Dutch color standard. Certain countries still retain this standard. The Dutch Standard is a series of sugars ranging in color from a very dark brown, numbered 7, to an almost pure white sugar, numbered 25. These samples are prepared annually in Holland. They are supplied to the sugar trade in small square bottles of uniform glass and size. Tropical 96° sugar usually falls below No. 16 of this standard.

**140. Classification of White Sugars.**—The classification of the sugars produced by the American refineries is given on page 180. The plantation product is usually called Plantation White or Plantation Granulated. Java white sugars in order to meet the market specifications of the East Indies must grade above No. 25 Dutch Standard.

**141. Warehousing of Raw Sugars.**—The full specification of a suitable warehouse is difficult to make. It is certain that the warehouse should be large and dry. There is difference of opinion as to whether it should be almost hermetically sealed during storage or open for free ventilation. The warehouse should certainly be closed on damp days on account of the deposition of moisture on the sugar with the reduced night temperature. Ventilation on dry days is undoubtedly beneficial.

For calculating warehouse capacities it should be remembered that a 325-pound bag of sugar occupies approximately 6.5 cubic feet of space and covers a floor-space area of 7 square feet when lying flat.

The subject of the deterioration of raw sugars in storage is dealt with in the subsequent chapter.

<sup>3</sup> *Loc. cit.*



## CHAPTER XIV

### KEEPING AND REFINING QUALITIES OF RAW SUGARS

**142. Introductory.**—Recognition of the importance of the physical and chemical characteristics of raw sugars other than the polarization has been increasing during the past decade. Formerly a sugar approximating 96 test was generally the only aim, but to-day the best controlled factories strive for a sugar which will not deteriorate in storage and which will work well in the refineries. The prevention of loss of polarization and weight during storage and transportation is of great economic value both to the manufacturer and refiner; and although the working qualities of the sugar in the refinery interest the refiner more directly, the manufacturer is also concerned in order to avoid discrimination against his product.

#### KEEPING QUALITY

**143. Control Laboratories.**—The first attempt to regulate the character of the raw product systematically was undertaken in 1914 by Dr. Spencer at the Central Control Laboratory of the Cuban-American Sugar Company at Cardenas under the immediate direction of the writer. (G. P. M.) Details of the methods used at Cardenas have been described by Tillery.<sup>1</sup> Later the Hawaiian Sugar Planters Association instituted a similar control and other sugar companies in Cuba and Porto Rico have since followed the same general plan.

**144. Desirable Characteristics.**—In order that a sugar will not deteriorate in storage it is generally agreed that it must have the following characteristics: (1) Be fairly free from insoluble matter; *i.e.*, boiled from a well-clarified juice. (2) Have a hard, uniform and fair-sized grain free from "rolled grain" or conglomerates. (3) Have a moisture content in relation to polarization to conform with certain "factors of safety." (4) Be an "unwashed" sugar; *i.e.*, the crystals must be surrounded by their original film of molasses. (5) Be manufactured under sanitary conditions to reduce contamination by fungi, yeasts, and bacteria to a minimum.

**145. "Cleanliness" of the Sugar.**—The direct bearing of "cleanliness" on keeping quality might be hard to establish but sugars boiled from poorly clarified juices would seem more likely to deteriorate. Particles of bagacillo and other organic matter hold moisture and serve as breeding places for micro-organisms while colloidal matter probably affects the formation of sharp hard grains in the pan-boiling.

<sup>1</sup> The Planter, December 3, 1921.

As a rapid test, dissolve a weighed amount of the sugar in hot water, shake and compare the suspended matter against standard tubes. A convenient method of reporting is on a scale of ten, No. 10 containing no suspended matter, No. 9, 20 mg. per 100 grams of raw sugar and so on up to 180 mg. per 100 grams for a No. 1 sugar. A weekly check may be made by determining the "insoluble matter" by weight. This is done by dissolving 20 grams of the raw in 200 ml. of water, bringing to a boil, filtering through a tared alundum crucible, washing with hot water to remove all sugar, drying at 105° C. and weighing. The result in milligrams multiplied by 5 gives "insoluble matter per 100 grams raw." Any sugar containing less than 40 mg. insoluble is extremely clean (No. 8 on cleanliness scale), while above 100 mg. is poor and marks the clarification of the factory as deficient.

**146. Character and Size of Grain.**—The size and general character of the grain have several important functions in connection with deterioration. A uniform, fair-size grain will purge more freely in the centrifugals, making it easier to attain the proper moisture-sucrose ratio for safety. The larger grain presents less surface and will therefore absorb less moisture during adverse storage conditions as conclusively proved by Owen,<sup>2</sup> while "rolled grain," conglomerates or grain clusters will occlude dilute molasses that may serve as an incubator for bacterial and mold growth.

The size, regularity and formation of the grain can be examined by means of the projectoscope, an improved form of which is shown in Fig. 42. It consists<sup>3</sup> of a Bausch and

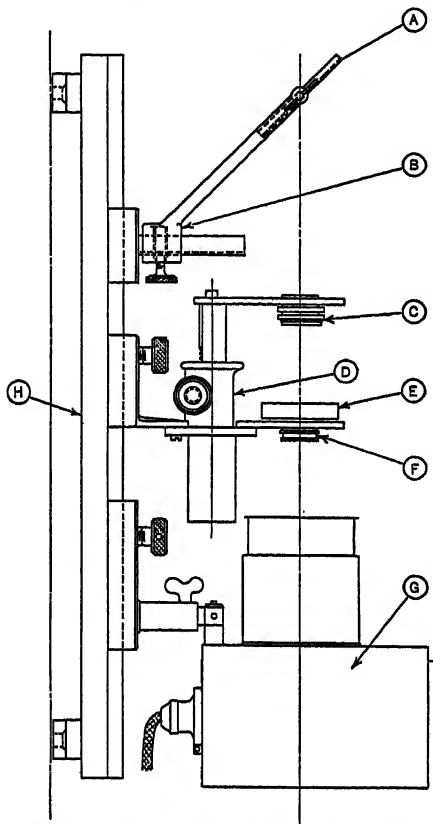


FIG. 42.—Projectoscope for Examining Sugar Crystals.

Lomb Micro-Tessar Lens, 72 mm. focus (C) mounted in a rack and pinion combination (D) with suitable condenser (F). The light source is a 6-volt 108-watt Mazda lamp in housing (G), the whole being mounted on an

<sup>2</sup> The Planter, Vol. LXX, p. 68 (1923).

<sup>3</sup> Meade. Ind. Eng. Chem., Vol. 13 (1921), No. 8.

"optical bed" (*H*) with a 45° mirror (*A*) at the top to direct the projected image horizontally. A screen (preferably a plaster of paris plaque), is placed 73 cm. away to give a magnification of 10 diameters.

A small amount of the sugar to be examined is placed in a Petri dish (*E*), covered with sugar-saturated alcohol, the grains being separated by rubbing gently with the ball of the finger. The image of the sugar on the screen may be compared with a series of grill-like squares drawn on the screen itself, giving the average size of the grains on a scale of ten. A No. 3 sugar is 0.4 mm. square; No. 4 is 0.6 mm.; a No. 5 is 0.8 mm. and so on. Most sugars fall between a No. 3 and No. 7, so squares representing these five sizes only need be drawn on the screen, multiplied by ten to allow for the magnification.

"Hardness" of Grain.—Hardness of the sugar crystal is difficult of determination; in fact some persons maintain that all crystals must be of the same hardness.<sup>4</sup> The recent work of Paine and Balch<sup>5</sup> shows that colloids and ash constituents of pan sirups were distributed throughout the volume of individual sugar crystals. Such crystals might easily be more friable ("softer") than a pure crystal. At any rate it is well recognized that many sugars feel soft when the crystals are rolled between the thumb and first finger, while others have a sharp, hard feeling and resist crushing to a greater extent. It may seem that a numerical expression of this characteristic involves too great a personal error, although it has been proved that two trained observers can check with practice.

147. Moisture Content and the "Safety Factor."—The importance of moisture content as an influence in the deterioration of sugars is now undisputed and it is agreed that the moisture percentage must be closely controlled. (The Spencer Oven (248) is particularly well adapted to moisture control of sugars.) The relationship between the percentage of water and the non-sucrose has been shown to be the determining factor, since the micro-organisms which cause deterioration cannot work in solutions of high density. The Colonial Sugar Company of Australia were the first to state the limits of this relationship in a widely known "Safety Factor" which prescribes that the moisture must not exceed one-third of the non-sucrose; or expressed numerically

$$\frac{\text{Moisture}}{100 - \text{Polarization}} = .333 \text{ or less.}$$

Many investigators have studied this point and they agree that this "factor of safety" is too high for many sugars and that to cover all cases the relationship should not exceed one-fourth; that is for positive safety

$$\frac{\text{Moisture}}{100 - \text{Polarization}} = .250 \text{ or less.}$$

These factors are calculated on the chart (Fig. 43) for polarizations ranging from 94.0 to 98.0. The use of the chart is simple. On the vertical scale at

<sup>4</sup> Norris. The Planter, Vol. 60, Nos. 4 and 5.

<sup>5</sup> Facts About Sugar. June 12, 1926.

the left-hand note the polarization of the sugar, and on the horizontal scale at the bottom the moisture. Follow the horizontal line out from the polarization until it crosses the vertical line from the moisture. If the lines cross to the right of the heavy line marked "Safety Factor of .250" the sugar will be almost sure to keep in storage; if it falls between the two lines it will probably keep provided all considerations such as warehouse conditions, character of grain, degree of contamination with micro-organisms, etc., are favorable;<sup>6</sup> <sup>7</sup> whereas if the lines cross to the left of the lighter line marked "Safety Factor of .333" it will almost certainly lose in polarization if stored for any length of time. It is obvious that the greater the failure to conform to the safety factor the more rapid and severe the deterioration is likely to be.

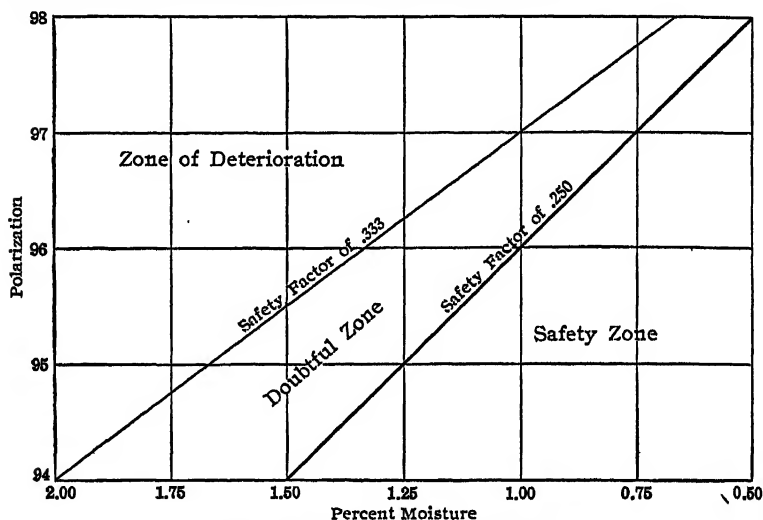


FIG. 43.—"Safety Factor" for Raw Sugars.

As an example of the use of the chart: A sugar polarizes 96.2 and has a moisture of 0.90 per cent. It is within the "safety zone." If the moisture of a sugar of the same polarization were 1.05 per cent it would fall in the doubtful zone whereas a moisture of 1.35 per cent at 96.20 would definitely mark the sugar as likely to deteriorate.

Browne<sup>8</sup> has shown that a mixture of a "safe" sugar and one having too high a moisture ratio may show a "safe" average but the crystals too high in moisture will deteriorate and become foci of infection for other crystals.

"Washed" Sugars.—The safety factors outlined in the previous section depend upon the density of the molasses surrounding the crystals and are valid only for sugars carrying the original film of molasses: "The practice of wash-

<sup>6</sup> Browne, J. Ind. & Eng. Chem., p. 190, 1918.

<sup>7</sup> N. & L. Kopeloff. La. Bulletin No. 175.

<sup>8</sup> Loc. cit.

ing raw sugars deprives their films of the protective action of the original molasses."<sup>9</sup>

Owen and others have also noted that the factors are not dependable for sugars which have already undergone deterioration. So it is evident that the polarization and moisture determinations should be made as the sugar leaves the centrifugals in order that positive assurance of keeping quality may be had.

**Bacterial, Yeast and Mold Contamination.**—Extensive studies by many investigators, including Kammerling, Grieg-Smith, Owen, Browne, the Kopeloffs, Van der Bühl and others have demonstrated beyond question that the loss of sucrose in raw sugars during storage is due to the action of certain types of micro-organisms in the film of molasses surrounding the crystals. The ordinary soil bacteria, certain types of yeasts (*torulae*), and various mold fungi, as well as combinations of all three, have been shown to be the actuating influence in different cases. Owen<sup>10</sup> sums up the recognition of the various forms of infection as follows: "Bacterial deterioration occurs only in dilute molasses films and gives abnormal Clerget values. If a sugar which was dried to a proper moisture ratio when manufactured evidences this type of deterioration excessive exposure to moisture during storage is indicated. Increase of polarization and loss in reducing sugars suggests the action of *torulae*, in which case there may be no real sugar loss. Losses in polarization without moisture absorption are almost invariably due to mold fungi if the "safety factor" is within the 0.333 zone. In such cases a careful study of sanitary conditions in and around the centrifugal and packing departments may discover and prevent the recurrence of this costly infection."

When it was first discovered that deterioration in storage was due to the action of micro-organisms it was thought that bacteriologic methods would become as essential to sugar-house control as the chemical routine. It is now thoroughly demonstrated that good clarification, good pan boiling, proper moisture control and reasonably sanitary manufacturing conditions (particularly at the centrifugals) will produce a sugar that will not go off in test during ordinary storage periods, so that bacteriologic methods need not be made use of except in the rarest cases and then only by experts called in for the purpose.

## REFINING QUALITY

**148. General Considerations.**—The characteristics desired by the refiner are (1) a sugar that will "wash" well, *i.e.*, give a maximum yield of washed sugar of high purity with the use of a minimum quantity of wash water in the centrifugals, thereby assuring a relatively small amount of "raw sugar washings" or "affination sirup," (Sec. 158); (2) a sugar that will filter rapidly in pressure or bag filters; and (3) a sugar of such color that it will be decolorized readily during char filtration.

Besides these primary characteristics the refiner also prefers a sugar of low ash-glucose ratio because such sugars in general give higher yields of granu-

<sup>9</sup> Owen. Facts About Sugar, March 25, 1925.

<sup>10</sup> Facts About Sugar, June 30, 1925.

lated sugar, also a low sulphate content in the ash to avoid scaling evaporator tubes and to insure freedom from turbidity in final sirups. It is obvious that, other conditions being equal, the higher the polarization the easier a sugar is to refine, but polarization is not a criterion of refining quality.

While the above statement of desirable characteristics would be concurred in by practically all refiners, agreement as to methods for determining these characteristics would be difficult to obtain. Wayne <sup>11</sup> says "it is our belief that if the sugar conforms to certain specifications for color, grain and filtrability that most of the other considerations are automatically taken care of at the same time." Horne <sup>12</sup> outlines certain laboratory tests with a view of paralleling house conditions, such as washing in a small centrifugal; filtration through bag filter-cloth; and decolorization by means of powdered boneblack. Other refiners feel equally sure that only actual practice in the refinery will demonstrate the refining quality.

The producer of raw sugar looks at the question from a different angle entirely. His first concern is for keeping quality, after which he is anxious to please the refiner only insofar as it is commensurate with highest yield and economic factory methods. It is safe to say that the producer of a "clean" well-boiled sugar of medium-size grain and fair color, with low "dye value" (*i.e.*, good clarification) need not concern himself with any of the more elaborate tests listed below.

**149. "Washing Quality"**—*Size and Character of Grain.* The same considerations which make size and uniformity of grain desirable for good keeping quality hold for a sugar which will work well in the refinery wash-plant centrifugals. A hard, uniform, medium-size to large, crystal purges freely, presents less surface for the wash-water to act upon and in turn carries less molasses on its surface to be removed by the washing process. Conglomerates and fine grains form a mat in the centrifugals, do not drain freely and require more wash-water which dissolves the crystal more easily because of the greater surface presented, giving an excess of affination sirup. The methods of pan-boiling used in making the raw (see pp. 92-97) also influence the "washing quality" greatly. If the nucleus of the crystal ("seed grain") is dark colored this color will remain in the washed sugar and the resulting melted liquor will be dark colored also.

The size and character of the grain as determined by the projectoscope for keeping quality will also serve for the washing quality.

*Laboratory "Washing" Test (Horne).*—Mix 100 grams of raw sugar with 45 ml. water, shake to saturation and strain through small piece of cotton cloth. Pour this sirup, which should be 92 ml., into the laboratory centrifugal basket with 200 gms. of the raw sugar, mix, and spin until dry. The weight of washed sugar left in the basket, divided by 2 gives the percentage yield of washed sugar.

**150. Filtrability.**—*Importance to the Refiner.* Most refiners now add an inert filter-aid such as kieselguhr to the washed sugar liquor and filter through pressure filters; some few still use phosphoric-acid and lime defecation and bag-

<sup>11</sup> The Planter. August 15, 1925.

<sup>12</sup> J. Ind. Eng. Chem., Oct., 1918.

filters; while one or two have the Williamson Clarification System which uses phosphoric-acid and lime but no filtration. In general a raw sugar which works well in any one of these systems will work well in all of them, and the term "filtrability" is here used to include the defecation as well as the removal of the precipitate, no matter what the material or means used. The wide variation in the filtering quality of different sugars and its effect on costs has long been recognized, since a poor filtering sugar not only slows up the entire refining process but also requires more defecating material or filter-aid, both of which are costly.

*Colloids and Filtrability.*—Investigations as to the cause and the direct and indirect measurement of this quality have been numerous in recent years. The work of Walter E. Smith<sup>13</sup> and others in Hawaii and of Badollet and Paine<sup>14</sup> on Cuban raws have shown the close relationship between filtrability and colloids and the finer dispersoids. Smith showed a direct relationship between filtration rate (by the Elliot test) and "non-settling matter" which is the suspended matter not removable by filtration through fine linen, or by centrifuging. This "non-settling matter" Smith claimed to be largely cane-wax which the clarification had failed to remove. He further showed that the raw sugar solutions after filtration through the Elliot apparatus, *i.e.*, after the colloidal material had been removed—filtered as rapidly on refiltration as a refined sugar solution of the same density. R. H. King<sup>15</sup> failed to corroborate Smith's findings, particularly as to the importance of cane wax, his conclusions being that the cause of the retardation of the filtration rate is the presence of gelatinous colloidal suspensions, both inorganic and organic, which form a pasty mass on the filtering medium and stop the flow of the filtrate. These gelatinous suspensions in the raw sugar are mainly due to the poor clarification of the juice.

Badollet and Paine correlated the actual filtering qualities of a number of sugars in a large refinery, their colloid content by ultra-filtration, and their "dye test value" (a rapid method of estimation of colloids as described below) showing that the filtering qualities were inversely as the colloid content and the dye test value, *i.e.*, colloids or colloidal material are the cause of poor filtering quality.

*Elliott Filtration Apparatus.*—R. D. Elliot devised an apparatus which is described by A. A. Blowski<sup>16</sup> as follows:

"Each filtering unit consists of a leaf 4 inches in diameter, equipped with a coarse-mesh screen on each side, over which standard cotton filter-cloth is fastened. For each filter leaf there is provided a trough to hold the unfiltered solution, a bottle to receive the filtrate, and a short piece of vacuum tubing to connect the leaf to the filtrate bottle. Each filtrate bottle is connected to a vacuum manifold which in turn is connected to a vacuum pump.

"A 1500-gram sample of the raw sugar to be tested is dissolved in 1500 grams of water at room temperature (20° C.). Thirty grams of kieselguhr, or 2 per cent on the weight of sugar taken, is added to the solution and mixed

<sup>13</sup> The Planter. October 11, 1924.

<sup>14</sup> The Planter. December 25, 1926.

<sup>15</sup> The Planter. 79 (1927), 221, 242, 287.

<sup>16</sup> Facts About Sugar. August 8, 1925.

thoroughly. After passing through an 18-mesh screen the prepared solution is transferred to the unfiltered liquor trough in which the covered filter leaf has been placed. After allowing the solution to stand for two minutes without further agitation, the vacuum is applied in such a way that it increases to 26 inches in two minutes. With the aid of a mercury seal the vacuum is maintained at 26 inches for the duration of the test. At the end of 30 minutes the test is complete, the vacuum is broken and the filtrate weighted.

"The weight of filtrate in grams, divided by 2658 and multiplied by 100, gives the filtration efficiency of the sugar. The standard weight of 2658 grams of filtrate, representing 100 per cent efficiency, was adopted simply because it happened to be the quantity of filtrate obtained from the best filtering sugar available at the time the test was developed. It was found that a standard sugar is not necessary, provided that a uniform cloth is available and all other conditions of the test are maintained constant.

"It should be mentioned that the temperature must be kept uniform, as a difference of two or three degrees centigrade makes an appreciable difference in the result. The test is conducted at room temperature because of the difficulty of maintaining uniform high temperatures, particularly when using vacuum. This is a radical departure from operating conditions, but it adds greatly to the simplicity of the test."

The use of a refined sugar solution as standard has been adopted by most investigators who have used this apparatus. The filtration in the cold has been objected to as not giving results comparable with filtrability as found in actual practice.

151. "Dye Test."—This is a simple and rapid method for the approximate determination of the quantity of colloidal material in sugars and sugar solutions. The description here given is abstracted from the articles by Badollet and Paine of the Carbohydrate Division of the U. S. Bureau of Chemistry who devised the method.<sup>17</sup>

The flocculation of colloids may be accomplished by the addition of free ions with an electric charge the opposite of that of the colloidal particles, or by adding a quantity of oppositely-charged colloid sufficient to produce mutual electric neutralization. In general, the colloidal particles in sugar products are negatively charged. When a negatively-charged colloid is mixed with a positively-charged colloid in a certain proportion, a point of electrical neutrality is reached at which the charges exactly neutralize each other, frequently causing mutual colloidal flocculation, and producing a precipitate that settles out of solution. If at this so-called "iso-electric point" the liquid containing the suspended flocs is examined with an ultramicroscope fitted with a cataphoresis apparatus, no movement of the colloidal particles or aggregates toward either electrode is observed. (Cataphoresis is the migration or progression of colloidal particles in a solution under the influence of an electric current.)

The cataphoresis apparatus consists of a U-tube, the center portion of which is capillary, and which has a platinum electrode in each arm (Fig. 44). The apparatus is fastened rigidly under the microscope, through which the motion of the colloidal particles in the capillary is observed. When the colloidal solution has been mixed with a portion of the solution containing the

<sup>17</sup> Int. Sugar Journal, Vol. 28 (1926), pp. 23-28, 97-103, 137-140.

The Planter. Vol. 79 (1927), No. 7.



oppositely-charged colloid, it is poured into the cataphoresis cell, and an electric potential is impressed on the electrodes. If the colloids present in the solution are not at a point of electrical equilibrium, they will progress toward the positive electrode if their charge is negative, and toward the negative electrode if their charge is positive. If they are at the "iso-electric point," the point where the electrical charges are exactly neutralized, they will show no progression toward either electrode.

The electric charges of the colloids in sugar liquor may be neutralized by any of several dyes, night blue being the one used most extensively, due to the fact that it produces the most rapid flocculation and a precipitate that settles most rapidly. In making the test 1 gram of the solid dye is dissolved in dis-

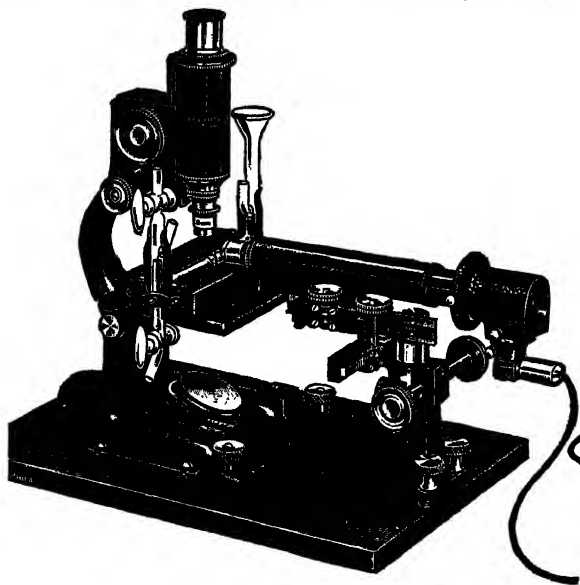


FIG. 44.—Dye Test Apparatus.

tilled water, and made to the mark in a liter flask. The solution to be tested should be first adjusted to some standard pH value. This standard is set at 5.2 in the case of raw cane juice and defecated juice, and at 6.0 in the case of other raw house and refinery products. The pH of the solution to be tested should always be recorded, since the dye number varies markedly with pH.

The "dye number" is calculated as follows: The weight of dry solids used in the test is determined, either by weighing beforehand, as in the case of raw and washed sugars, or by measurement and the use of the specific gravity or the refractive index. Then the number of milliliters of dye solution necessary for neutralization of the colloids in the sugar solution, multiplied by 100, and divided by the weight of dry solids used in the test, will be the "dye number" of the substance in question.

## 146 KEEPING AND REFINING QUALITIES OF RAW SUGARS

**152. Technic of the Dye Test.**—*Procedure No. 1 for Raw and Washed Raw Sugars.* Five grams of the sugar are dissolved in 25 ml. of distilled water, and the solution is filtered through a 100-mesh screen, which is then washed with small quantities of water. The filtrate and washings should be combined and diluted to a volume of 100 ml. in a 600 ml. beaker. A raw sugar solution prepared with distilled water will generally have a pH value of about 6.0, and adjustment of pH is not usually required. If, however, the pH value is found to vary from 6.0 by more than 0.2 or 0.3, the pH of the solution should be adjusted to the value 6.0 in order that all raw sugars examined may be on a comparable basis so far as influence of pH is concerned. The pH of the raw sugar liquors may be adjusted by the addition of small amounts of 0.05 normal sodium hydroxide or hydrochloric acid and tested separately with an indicator solution in any convenient comparator.

Ten milliliters of the standard dye solution is added to the 100 ml. of sugar solution, which is then tested in the ultramicroscope. If the colloid particles move toward the positive electrode they still carry a negative charge, in which case the stopcocks are opened and the solution is drained into the original beaker, more dye solution is added, and after mixing well, the sugar solution is retested in the cataphoresis cell. This process is repeated until the colloid flocs fail to move progressively toward either anode or cathode when current is applied at the electrodes.

For example, suppose that the sugar solution required 20 ml. of dye solution for adjustment to the neutral or isoelectric point (zero progressive movement of the colloid flocs). Then

$$\frac{20 \times 100}{5} = 400, \text{ the dye value of the sugar tested.}$$

As a check on the electric neutralization of the colloid particles, add a small excess of the dye after the neutral point is reached. This will reverse the electric charge in the colloid particles, causing them to become positively charged and to move toward the cathode. The volume of dye solution used in the calculation of the dye value should, of course, be that which *just* causes electric neutralization.

Procedure No. 1 is sufficiently accurate with raw sugars of a moisture content as high as 2 per cent, but for sugar of a higher moisture content it is preferable either to dissolve the sugar to a 10° Brix solution and use Procedure No. 2, or to weigh out 5 grams of the sugar, as in Procedure No. 1 and correct for its moisture content.

It is advisable to wash a portion of each sample of raw sugar examined to 99 purity and to determine the dye value of the washed sugar as well as that of the original sugar. The purpose of this scheme is to ascertain the relation between the quantity of colloids eliminated by washing and the quantity remaining in the washed crystals. This step is important in view of affination of the raw sugar before filtration and subsequent treatment of the melt. Raw sugars of good refining quality when washed to 99 purity give a dye value of approximately 100 or less, whereas some raw sugars of poor refining quality when washed give dye values well over 100—sometimes as high as 200.

*Procedure No. 2 for Raw Cane Juice, Defecated Juice, Filter Press Juice, Massecuites, Sugar, Sirups and Molasses.*—The dye value of the raw cane juice, defecated juice, and filter press juice may be determined at the original density. But for massecuites, sugars, sirups, and molasses, considerable time may be saved by diluting an aliquot of the double diluted laboratory sample to 100 ml. for the dye test. \* Use distilled water. Weigh out a definite quantity of diluted liquor using a sample of such size that 15–25 ml. of the dye solution is required for neutralization and base the dye value calculation on the percentage of solids in the weighed sample. The percentage of solids in the weighed sample can be calculated with sufficient accuracy from the refractive index or specific gravity by hydrometer. The pH may be adjusted by the addition of 0.05 normal hydrochloric acid or sodium hydroxide if required; 5.2 being a convenient pH for raw and defecated juice while 6.0 is best for other sugar-house products.

After the pH is adjusted (if required) 10 ml. of dye solution is added and mixed thoroughly with the 100 ml. of liquor, after which the test is carried out as in Procedure No. 1.

**153. Miscellaneous Notes on the Technic of the Dye Test.**—1. The voltage across the cell should be around 200 volts. This can be easily procured by a battery of five forty-five volt radio "B" batteries. A 50-watt lamp should be connected in series with the cell. The switch controlling the voltage across the electrodes should be of the reversible type, in order to eliminate polarization.

2. Adjust the microscope carefully, so that it is focused at a point in the vertical central plane of the capillary at a distance of 0.293 times the radius of the capillary below the upper wall of the capillary. This focusing may be best accomplished by the following method: First see to it that the microscope is in the central plane of the capillary, and then focus on the upper wall of the capillary by using a suspension of powdered granulated sugar in sugar-saturated alcohol, and focusing rapidly on the highest visible particle before the suspension has had the time to settle. Then allow the suspension to settle, and after recording the reading of the vernier on the milled head, the microscope is focused on the lowest particle in the capillary. One-half the number of turns on the milled head (expressed to 100ths) necessary to change the focus of the microscope from the upper to the lower wall is multiplied by 0.293, and after re-focusing on the upper wall of the capillary, the microscope is turned down the calculated fraction of a turn.<sup>18</sup>

3. Keep the stop-cocks of the cataphoresis cell well greased.

4. In filling the cell, be sure that the liquid overflows at the top stop-cock so that no air will be retained in the closed chamber.

5. In making a test, be sure that the stop-cocks are well closed.

<sup>18</sup> In some cases, where the microscope is equipped with an objective of 8 mm. or less focusing distance, it will be impossible to focus on the lower wall of the capillary, in which case it will be necessary to determine the radius of the capillary in terms of turns of the fine adjustment of the microscope by means of an objective of greater focus (for instance 16 mm.), and then focusing on the upper wall with the higher-powered objective and lowering it the calculated amount.

6. When the colloidal particles are observed through the microscope (after the instrument is correctly adjusted), they should not show any *progressive* movement (neglecting brownian movement) toward either electrode when the current is not applied. If the particles continue to move when the current is off, look for a leak in the stop-cocks or for air-bubbles in the closed chamber or for cracks in the glass. The colloid particles must show no progressive movement whatever before making a test.

7. Always mix the dye solution and sample thoroughly before making a test in the cataphoresis cell. Try to standardize the test as much as possible, regarding original amount of dye added, number of milliliters added in successive fractions, time of stirring and standing, etc.

8. The cell should be cleaned every few days. Alcohol or dilute hydrochloric can be used satisfactorily, but it is necessary to remove all traces of the cleaning substance before another test is made.

**154. Color of Raw Sugars.**—The amount and character of the color of a raw is of obvious importance to the refiner. The factors which influence color of raw sugars are the character of soil supplying the factory with cane, the ripeness of the cane, the clarification of the juice (particularly as to the avoidance of excess lime salts), the methods of pan-boiling (see page 92), and the prevention of overheating and caramelization throughout the process. T. B. Wayne<sup>19</sup> made an extensive study of adsorption of coloring matters in sugar products by boneblack, using spectrophotometric color analysis. He found that the reddish color due to the caramelizing action of lime and heat on invert sugar is not necessarily the most difficult type of color to remove, as has so long been supposed by refiners. This red coloring matter is less easily removed when small proportions of char are used, as in most laboratory tests, but when the ratio of char to liquor is increased the adsorption curve of the reddish sugars continues in a straight line to almost complete removal, whereas for greenish-gray sugars the initial color removal is greater for small char-ratios but falls off when the ratio of char to liquor approaches that of actual refinery conditions (60 per cent to 100 per cent). In other words the residual color after intensive char-filtration will be greater with greenish-gray sugar (color due to colloidal iron) than with reddish sugars (color due to incipient caramelization). The resulting refined sugars from the liquors in the first instance will have an ugly dead gray appearance.

A complete discussion of color-determination methods is given in Chapter XXIII and the methods for raw sugars on page 337 of the chapter on Analysis of Sugars.

<sup>19</sup> Ind. Eng. Chem., Vol. 18 (1926), No. 8.

## CHAPTER XV

### SUGAR REFINING

**155. Introductory.**—The process of cane-sugar refining is simple in theory, but very complex in actual practice. The working up of the lower-grade materials, the handling of the sweet-waters and the control of the bone-char filtration involve an enormous amount of detail, much of which cannot be fully considered in this chapter for reasons of space.

The narrow "margin," or difference between the price of raw and refined sugars, has tended to cause the concentration of the industry into large units. Shipping facilities and the multiplicity of grades of refined sugar have also helped in bringing about this concentration. Practically all the refined sugar used in the United States is now produced in about 20 large refineries situated in the principal seaports.

Until very recent years a traditional conservatism on the part of refiners prevented the publication of investigations relative to the industry. This dearth of literature on refining has led to the false impression among those engaged in other branches of sugar work that the process was "cut and dried" and devoid of scientific interest, but during the last few years many articles by refinery men, particularly on the subject of char filtration, have appeared in the scientific journals. A reference book called "The Elements of Sugar Refining" by Bardorf and Ball<sup>1</sup> has also been published.

**156. Definitions.**—Formerly refineries were divided into two classes; those which produced only the pure white granulated and its modifications, and those which made both the granulated sugar and "softs," or the soft-grain, white and yellow sugars of lower purity. "Soft-sugar" refineries, as the latter class were called, generally melted raws of a lower test and had larger char-filter capacity than the "hard-sugar" houses, but this distinction has now entirely disappeared. This change has come about in part through the decrease in the demand for soft sugars and also because most of the raw sugars now available for refining are of 96° polarization or above. Furthermore, the refiners found that many of the expedients used by the "soft sugar" refineries for obtaining low test liquors of bright color were uneconomical and that similar results could be secured by the more economical methods used in the "hard sugar" plants.

The term "liquor" is used in refining to refer to a heavy sugar solution from which no sugar has been removed by crystallization; that is, since its last treatment or char-filtration. "Sirup" refers to a heavy solution from which sugar has just been crystallized, and corresponds to the "molasses" of the

<sup>1</sup>Chem. Pub. Co., 1925.

raw factory. A "sweet-water" is a wash-water which contains sufficient sugar to warrant recovery. A "magma" is a mixture of crystals and sirup—the massecuite of the raw house. The term massecuite is also very generally used in refining. Dissolving is described as "melting" the sugar, and the amount of raw sugar handled in twenty-four hours is termed the "melt."

**157. Raw Materials.**—Centrifugal sugars of 96° test or above form by far the larger proportion of the melt of the refineries. The present tendency, however, is for the raw-sugar factories to produce sugars of higher test than 96° with a view to obtaining better storage qualities. The lower-grade sugars, cane seconds or molasses-sugars, muscovadoes, Philippine mattes and concrete-sugars, palm-sugars, and others are now produced in such small proportion that little consideration need be given them by the refiner.

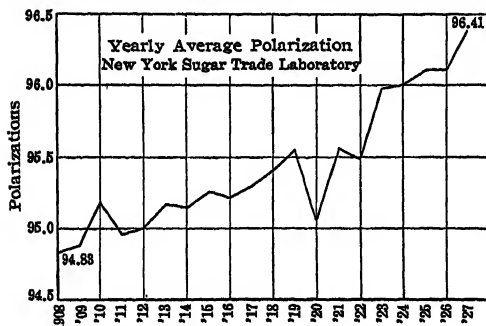


FIG. 45.—Average Raw Sugar Polarizations.

shown in the chart, Fig. 45. A comparison of the distribution of the various grades for 1914 and 1927 is as follows:

Sugar Testing	Per Cent of the Total Number of Samples	
	1914	1927
Above 98°	0.924	0.497
97-98	9.453	15.082
96-97	38.602	65.313
95-96	31.037	15.602
94-95	8.041	2.643
93-94	3.005	0.205
92-93	1.009	0.229

It will be seen that in 1914 less than 50 per cent of the samples were above 96° test, whereas in 1927 about 81 per cent were above this grade. It is highly probable also that the major portion of the samples between 95 and 96 were

deteriorated sugars originally made above 96 test. These figures all show the tendency towards high-test raws and also the universal practice of making one grade of sugar only. (See p. 90.)

The raw sugars arrive at the refinery in various kinds of packages. Jute-bags containing 300 to 330 pounds each are received from the West Indies. Hawaiian and Central American sugars are packed in smaller jute-bags. Java sugars are packed in palm-leaf baskets. Formerly barrels and hogsheads were used for muscovadoes and concrete-sugars. Each package of sugar is weighed and sampled before it is dumped.

The raw sugar is dumped into bucket-elevators and is carried to the minglers. Most of the larger refineries have recently installed storage-bins for raw sugars which are placed above the minglers. The packages of sugar are hoisted directly from the ship and after weighing and sampling are emptied into the bins. Sufficient sugar is dumped during the day to supply the refinery for the twenty-four hour period and the sugar is fed continuously by scroll conveyors from the bins to the minglers. This method of storage materially reduces the cost of handling the raw sugars.

The empty jute-bags are washed with hot water for the recovery of the adhering sugar, dried and sold. The wash or "sweet-water" is mixed with other similar solutions and is evaporated to a sirup and thus enters the manufacture. The palm-baskets are steamed and then burned under the boilers in specially constructed furnaces. Hogsheads and barrels are steamed and sold in the rare cases that these are received.

**158. Affination. Washing the Raw Crystals.**—The first step in the process of refining is termed "affination," or more commonly "washing," and consists of removing the adhering film of molasses from the surface of the raw sugar crystal. The molasses film has a purity of 65 or lower, depending on the class of massecuite from which it was boiled in the raw factory, while the crystal itself is nearly pure sucrose. The separation is accomplished by mingling the raw with a heavy sirup (65°–70° Brix) and then purging the mixture in centrifugals, washing with water after the sirup has been spun off.

The raw sugar enters the minglers, which are heavy scroll conveyors fitted with strong mixing flights, and is then mixed with water at the beginning of the run to form a heavy cold magma or mash which drops into a mixer of the same style as those used in raw sugar work. With the mingling and the mixing the added water dissolves the molasses film (and a small part of the sucrose of the crystal) and forms a heavy sirup. The magma is then purged in centrifugal machines, the self-discharging type, either belt-driven or electric, being generally used for "wash-plant" work in American refineries. After the sirup has been spun off, the sugar in the centrifugal basket is sprayed with water (6–10 quarts per 40-inch machine) and the washed sugar is dropped from the machine to be sent to the melter as described below. The affination process if properly conducted should yield a light-colored washed sugar of close to 99 coefficient of purity.

The sirup purged from the mash is returned to the minglers for mixing with the incoming raws instead of water. It is evident that with the successive minglings and purgings the "raw sugar washings" (also called "wash sirup")

and "raw greens") will become lower and lower in purity and will increase in amount much beyond the needs of the mingling process. That portion of the raw sugar washings not used for the mingling is heated and diluted to about 54° Brix with dark sweet-waters, after which it is pumped to the defecators. In order to reduce sugar losses through material being on hand too long, it is usual to start the mash with fresh water once every twenty-four hours, all the washings on hand at that time being pumped to the defecators. The portion of the washings used for mingling (called "sirup for washings") is diluted in some refineries to 65° Brix or is warmed to 120°-125° F., or both, the object being to reduce the viscosity and facilitate the removal of the dense molasses film on the raw crystals. No control, other than appearance, is kept on the character of the mash from the minglers, the only requisite being a good mixture of such a consistency that it will purge rapidly.

The "raw sugar washings" vary in purity from 74° to 80° and constitute from 14 per cent to 18 per cent of the weight of the raws melted (after dilution to 54° Brix in the defecators). The amount of raw sugar washings will be greater as the test of the melt is lower. The character of the grain (Sec. 149) will also affect the amount; a small, soft-grain sugar giving more washings than a well-boiled large-grain raw.

When very small, soft-grain low-grade sugars, such as muscovadoes and concretes, are handled, it is customary to melt them directly without washing. The resulting low-grade liquor is defecated and treated exactly the same as the washings. The "washed sugar" is dissolved in about one-half its weight of water in a tank provided with mixing-arms called a "melter," exhaust steam being applied from a perforated coil to aid solution. High-test sweet-waters, particularly those from pressure filtration, are used in melting, but it is obviously bad practice to contaminate the melted liquor with impurities by using other sweet-waters which are much below the purity of the washed sugar itself. Maintaining the test of the washed sugar liquor close to 99 purity at all times aids the defecation and improves the grade of the char-filtered liquors.

#### DEFECATION

**159. General Principle.**—The raw washed sugar liquor from the melter contains some insoluble material, such as bagacillo, clay, sand, etc., and an appreciable amount of fine suspensions and dispersoids as well as gums, pectins, and other true colloids that have escaped the clarification of the raw-house or have been formed in the subsequent process of manufacture. The raw liquor is also acid as a rule. Similar considerations in a more exaggerated form hold for the raw sugar washings (affination sirup), which contain the major portion of the impurities of the original raw. Defecation may be defined as the treatment of the raw liquor or raw washings with certain substances and heat in order to render the solution suitable for filtration or clarification.

A great number of defecants have been suggested or experimented with in refining work. All have essentially the same idea, namely, the formation of a flocculent precipitate during heating to adsorb the colloids and enmesh the suspensoids and suspended matter so that they may be caught and removed



by the meshes of a cloth or fabric. Blood or blood albumen was the defecant formerly used but its use was entirely discontinued some thirty or forty years ago.

The defecation, no matter in what form, is carried out in defecators (or "blowups" as they are generally called in refining practice) which are usually circular tanks with conical bottoms to facilitate drainage and cleaning, fitted with steam coils and air connections for agitation.

The raw sugar washings are defecated in separate blowups, in the same manner as the washed sugar liquor, the difference being in the quantity of defecant used. Many refineries also defecate other materials returned from the process—remelt sugars and granulated sirups—which are to be char-filtered.

**160. Phosphoric Acid-Lime Defecation.**—Up to 1915 the defecating materials most commonly employed were milk of lime and phosphoric acid. This form of defecation is still essential in those few refineries which employ bag-filters and also in conjunction with the Williamson System (164). Inert filter-aids and pressure filtration have superseded the phosphate-lime defecant, however, in the majority of the plants of the United States. The phosphoric acid may be in the form of monocalcic phosphate, phosphoric acid paste or a clear phosphoric acid.

The monocalcic phosphate may be made in the refinery by the action of commercial hydrochloric acid upon boneblack. This is called "black paste," and contains from 10 to 12 per cent of available phosphoric acid ( $P_2O_5$ ). The black paste contains chlorides and these are not adsorbed by the char, hence increase the quantity of residual sirup. Phosphoric acid paste may be made in the refinery by treating boneblack dust with sulphuric acid. Pastes of this type may be found in the market under various commercial names. They are apt to have a high content of sulphates which will be largely retained by the char in the filters, increasing the sulphate content of the char. The clear phosphoric acid is made by treating boneblack dust with sulphuric acid and then filtering out the solids in wooden-frame filter-presses. The soluble sulphates are reduced to a minimum by diluting the phosphoric-acid solution to 15° Brix.

Phosphoric acid pastes are now on the market which are of high purity, free from objectionable salts and are cheaper, per pound of  $P_2O_5$ , than the home-made materials. The refineries employing phosphoric acid now generally purchase the material in this form. A paste of this variety electrolytically produced showed the following analysis:

Per Cent $P_2O_5$ .....	49.97
CaO.....	12.38
Iron and alumina.....	.54
$SO_3$ .....	.43
Arsenic (Parts per 1,000,000).....	.2

The lime is introduced as a milk of about 20° Brix. The phosphoric acid is first added and the lime immediately afterward, usually in sufficient quantity to give a faintly alkaline reaction to litmus (pH 7.0–7.3.) The amount of

The defecated liquor is run on to the top of the filter, which is surrounded by a shallow curbing, and flows down through the bags, the precipitate being retained. The filtered washed-sugar liquor is of a light-brown color, quite free of suspended matter or turbidity. The filtered raw-washings are also clear but of much darker color. After from fifteen to twenty hours' use the bags fill with mud and filtration becomes very slow. The liquor going on to the filter is now shut off and the liquor remaining in the bags is sucked out by a vacuum-pipe, or is allowed to drain out. The filter-bags are flushed once or twice with thin alkaline sweet-water from the filter-presses and the mud is further washed in the bags, as they hang in the filter-casing, by introducing a jet of hot water into each successively. This "sticking" is repeated from three to five times, after which the bags are taken out of the filter and washed in a series of tubs to remove all the mud. The bag-filter sweet-waters are sent to the evaporator for concentration. The mud-water, *i.e.*, the mud and water from the tubs, is limed, diluted and filter-pressed. The press-cake is discarded or sold for agricultural use, and the filtrate, press-water, is used partly in flushing filters as mentioned above, and the remainder is evaporated to a sirup.

**163. Pressure Filters.**—These are filters in which the cloth or filter fabric is held on a frame with sufficient backing so that the liquid may be forced through under pressure from the outside to the inside of the filter element. The pressure generally ranges up to 30 to 40 pounds per square inch. As already explained pressure-filtration involves the use of inert filter-aids such as kieselguhr or paper-pulp, since the tricalcic phosphate precipitate will not permit of any flow under pressures beyond the few pounds of hydrostatic head encountered in the bag-filter.

Very recently, the use of a small proportion of phosphoric acid and lime in conjunction with the kieselguhr or paper pulp has been put into practice. This has been resorted to in order to obtain the removal of colloidal iron coloring matter (polyphenols of iron). These iron compounds impart a dark greenish-brown color to the washed sugar liquor which the phosphate precipitate removes. The principle in all pressure filters is the same, the defecated liquor containing the filter-aid being forced through the press by centrifugal-pump pressure. The cake is retained on the cloth and the clear liquor flows through and emerges from the filter element which is of the leaf-type in most of the filters used. The cake is then washed free of sugar in place with hot water and sluiced off the plates by suitable means, or it is sluiced off after partial washing, the cake then being mixed with water and refiltered. The latter practice is safer as it reduces the possibility of sugar losses.

**Kelly Filter-press.**—The Kelly was the first filter of this type to be used in cane-sugar manufacture but it has been entirely replaced by other types. Filter-bags are placed over frames suspended upon suitable pipe-racks and are enclosed in an inclined cylinder. Each bag communicates with a filtered juice-canal. Treated liquor is admitted to the cylinder under considerable pressure. The suspended matter attaches itself to the cloths and the liquor flows through the latter into the pipes leading to the canal. The operation is stopped from time to time and the end door of the cylindrical body is opened for the removal of the rack and the discharge of the mud. The mud or

press-cake may be washed to low sucrose content in the press. The cloths require changing at very infrequent intervals.

*Sweetland Stationary Leaf-filter.*—This press, originally called the "clam-shell" type of press, took its name from its two parts opening and closing after the manner of the clam-shell steam-shovel bucket. The body of the filter (Fig. 46) comprises two semi-cylindrical members hinged together, with suitable gaskets, to form a water-tight cylinder when closed. The filter leaves are composed of crimped-wire screens, each provided with an outlet nipple for the filtrate. Filter-cloth is fastened over these screens and these latter are so arranged that they may be clamped tightly in place in the filter body with the outlet-nipples in connection with the delivery fittings outside the filter.

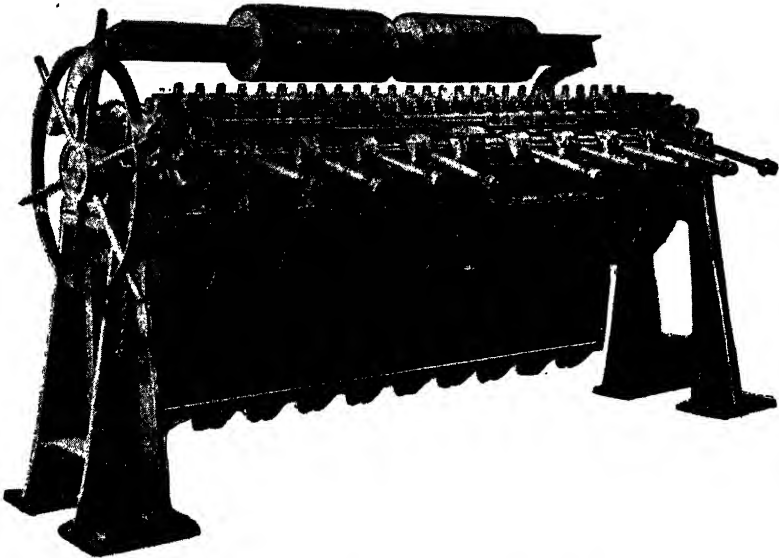


FIG. 46.—Sweetland Stationary-Leaf Press.

Variable spacing is provided for the leaves to suit different filtration conditions,  $1\frac{1}{2}$ - or 2-inch centers being used in refineries. Each leaf has separate delivery, shut-off cock, glass delivery-tube, etc. The opening and closing of the press is easily and quickly accomplished, the largest presses using hydraulic power.

The sluicing device consists of a manifold pipe passing through the entire length of the filter inside the shell just above the leaves (at the back of the figure). This pipe extends out through stuffing boxes at both ends of the filter, nozzles being placed at equal intervals along the pipe so that a nozzle comes directly behind each leaf. The stuffing boxes allow a double movement of the pipe: first, it may be rotated through an arc of approximately  $110^\circ$ , by means of a handle attached to one of the projecting ends; second, it may be moved longitudinally through the stuffing boxes during this rotation.

Water or other liquid is admitted under high pressure (approximately 80 pounds per square inch) and discharged through the nozzles, thus directing streams of water across the surfaces of the filter leaves and cutting down the cake. Provision is made for moving the pipe longitudinally as well as rotating it, making it possible for one nozzle to serve both sides of a filter leaf. A self-reversing sluicing mechanism combines these two movements of the pipe so that continued movement of the handle up and down first directs streams of water across one side of the leaf and then across the other side.

*Sweetland Cantilever Filter.*—This is a recently designed rotating-leaf filter shown open in Fig. 47. Each circular filter disk is composed of six independent sectors which facilitates drainage, as well as repair and replacement of filter

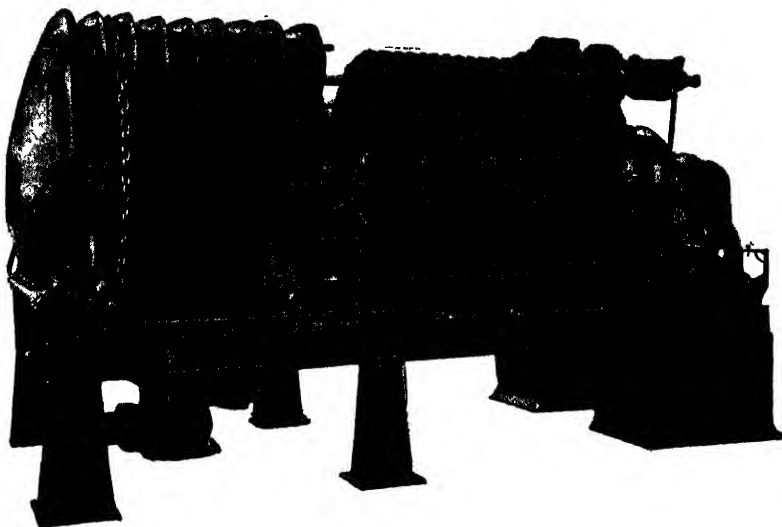


FIG. 47.—Sweetland Cantilever Press.

elements. The press operates at two speeds, the disks being rotated slowly during filtration and washing (or "sweetening off") and much more rapidly during sluicing. The sluicing mechanism is similar to that in the stationary leaf Sweetland. The advantages of rotating-leaf filters enumerated in the description of the Vallez Filter are valid in this case also.

*Vallez Rotary Filter.*—This was the earliest of the rotary-leaf filters to be used extensively. As shown in Fig. 48 it consists of a series of disk-shaped filter "leaves" mounted on a hollow shaft inclosed in a cylindrical cast-iron vessel or "shell." These leaves are hollow and are covered with filter cloth. The material to be filtered is pumped into the filter shell under pressure. The leaves and shaft revolve slowly as the liquid is being filtered through them. Each leaf filters as a unit, discharging the filtrate into the shaft, which serves as a common header for carrying the filtrate out of the shell. The Vallez is

generally used in sugar-refineries with woven-wire filter cloth of monel metal or bronze. This does away with the frequent changing of cotton cloths, to accomplish which in this filter necessitates the entire removal of the shaft and all filtering disks.

When the filtering cycle is complete the cake is sweetened off either partly or entirely and then the remaining water in the filter is agitated violently with air which loosens the filter mat on the leaves. A final spraying with water completes the cleaning after the cake and water have been discharged from the bottom of the filter.

The rotating leaf type of filter offers several advantages over the fixed type. The cake is more evenly deposited on the leaf; the filter-aid is kept in better suspension; breaks in the cake are less frequent and washing-off in place may be more efficiently accomplished. Finally, the removal of the cake from

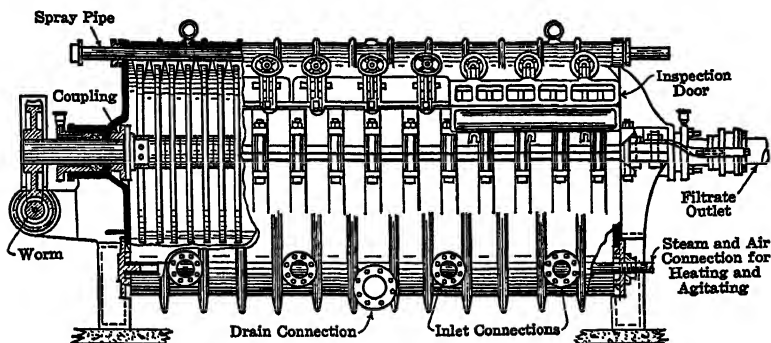


FIG. 48.—Vallez Rotary Filter.

the leaves is much more readily done by a spray since all parts of the leaf are brought into closer contact with the sluicing jets.

*Plate-and-frame Presses.*—It has recently been found that pressure filtration with filter-aids such as kieselguhr can be carried out in plate-and-frame presses of the type so generally used in the raw house (sec. 67) and refinery for filtration of muds. The operation is as efficient, so far as filtration is concerned, as with the leaf type of filter, but there is the labor of opening the presses, cutting down the cake, etc., which the closed presses do not require. Both washed sugar liquor and defecated washing are being successfully filtered with plate-and-frame equipment. Sweetening-off is said to be easily accomplished by washing in the press.

**164. Williamson Clarification System.**—This is a process patented by George B. Williamson, of Louisiana, for the clarification of defecated raw liquors without filtration. The system is continuous and makes use of the same phosphoric-acid and lime defecation that is used with bag-filters. Essentially, it consists of impregnating the defecated liquor with air and then heating to 210° F. in flat vessels so that the air bubbles will rise and carry with them the flocculated material, forming a scum which is drawn off by suitable means.

The defecation is carried out as described before except that the liquor is not heated in the blowups after the addition of the phosphoric acid and lime. The temperature at which the raw liquor comes from the melter (about 160° F.) is suitable. From the blowups the defecated liquor flows through aerators, which consist of small tanks containing jet nozzles, through which compressed air is blown into the liquor. The liquor leaves the aerators completely filled with air-bubbles and flows to the clarifiers, which consist of flat tanks 6 feet  $\times$  12 feet  $\times$  2 feet deep containing transverse copper steam pipes at the bottom with 100 square feet heating surface for each clarifier. The liquor entering the clarifier at one end flows slowly through it, the temperature rising until it is 210° F. when the liquor reaches the outlet end. By this time the air-bubbles have carried the flocs of precipitate to the surface forming a tough blanket of scum. This blanket is drawn off by means of a slowly-turning roller which pulls the scum over a lip and into the mud trough. The clear defecated liquor is drawn off through pipes from the body of the clarifier about 6 inches below the surface. Each clarifier has a capacity of from 800 to 1000 gallons of liquor per hour, a million pound melt requiring eight clarifiers for washed sugar.

The clarified liquor contains some flocs of bagacillo and other light material which are removed by passing the liquor through a woven wire metallic cloth screen 120  $\times$  120-mesh. The solids drawn off as mud amount to 5 to 7 per cent of the solids entering the clarifier.

The main advantage of the Williamson System is that it permits the use of phosphoric-acid and lime defecation (with the attendant removal of objectionable colloids and coloring matter) without the use of bag-filters, which are so expensive as to labor and objectionable because of lack of cleanliness and the possibility of sugar losses through the souring of sweet waters. The equipment is simple and requires little expense in upkeep and operation. The disadvantages of the system are several, though none of them are insurmountable in practice. The first objection is the heating of heavy sugar liquors to 210° F. with the possibilities of inversion or decomposition through overheating. In actual practice it has been shown that with a careful control of the reaction of the liquors and of the temperatures the losses are negligible. Any acid liquors will immediately cause heavy losses, however. Another objection is that the clarification is not as positive as filtration and that the effluent liquor will be variable in character. This is true to an extent but is not of great importance in practice if the test of the washed sugar is kept up to 99° purity. The process is unquestionably sensitive to raws of poor character, possibly more so than a filtration system. The Williamsons rarely give brilliant clarified liquor but the turbidity is due to particles of such a size that the char readily removes it and the suspended matter so removed does not tend to clog the char.

The most valid objection is that the Williamsons are not adapted to work on low purity liquors. With raw washings they will not serve at all because of the bulk of the precipitate set up by the defecation and even with remelt liquors of 94-96 purity they do not give very good clarification.

**165. Handling of Muds from Cloth Filtration.**—The mud waters from the bag filters, cloth filters or Williamson clarification are generally sent to mud blowups where they are diluted, heated and limed to a pH of 8.0 to 8.4, after which they are filter pressed in plate-and-frame presses similar to those used in cane factory practice. As has already been mentioned, the sluiced filter-cel from pressure filtration of washed sugar liquor is now quite generally reused in the defecation of raw sugar washings. When this is practiced the sluicings from the liquor presses are dewatered in Oliver vacuum filters and the cake is then puddled with water to a cream and added to the washings blowups. This results in an economy of kieselguhr. Oliver filters have been substituted in some refineries for plate-and-frame presses for filtering the final mud waters.

The high-test sweet waters are used for melting sugar as previously described while the dark-colored sweet waters go to the evaporator for concentration with other sweet waters.

**166. Control of the Defecation and Filtering Stations.**—The amount of defecant varies with the character of the raw sugar melted and with the test of the liquor to be treated. The determining factors in the amount to be used are the speed of filtration and the clarity of the filtered liquor. Experience and trial-and-error are the only guides in this matter. In general the lower the test of the melt the more defecant or filter-aid required but it has been shown (Chap. XIV) that filtrability is not strictly a function of the test of the raw; some raws giving less than half the speed of flow that other raws of the same test give.

The adjustment of the reaction of the liquors at the blowups is best done by hydrogen-ion (pH) control and most refineries employ this system. The usual pH range for filtered washed sugar liquor is 7.0–7.3 pH (faint alkalinity to litmus) but some refineries prefer higher alkalinities than this (7.5–8.0 pH). As will be shown later the changes in pH of the liquor during the char filtration determine what initial pH is chosen for the on-going liquor.

A method which has proved successful in the adjustment of the reaction of each blowup is outlined on page 28. Ordinary unskilled workmen have no difficulty in maintaining the pH within limits of .2 to .3. It should be noted that with the phosphoric acid-lime defecation if the pH is determined on defecated liquor containing the phosphate precipitate it will show from .3 to .5 pH higher than after filtration or clarification. In adjusting the reaction at the blowups allowance must be made for this drop in pH due to the removal of the phosphate precipitate. That this drop is due to the presence of the tri-calcic phosphate and not to any decomposition during the filtration or clarification can be proved in the laboratory by determining the pH of the defecated liquor before and after removal of the precipitate by filtration through carefully washed paper.

Blowski and Holven<sup>2</sup> in a careful study of the use of pH in the refinery showed that test papers and titrations are no reliable guide to the reactions of refinery liquors. To prove this point they made up several different refinery materials to definite total acidities and alkalinities by adding known quanti-

<sup>2</sup> Ind. Eng. Chem. Vol. 17 (1925), No. 12.

ties of  $N/28 H_2SO_4$  and  $N/28 NaOH$  to the neutral solutions. They determined the pH of these solutions at these known total acidities and alkalinities; some of their results being plotted in Fig. 49. It will be seen that an acidity of .003 in washed sugar liquor (raw liquor) gives a pH of 5.5, at which point active inversion would take place, while raw sugar washings at double that

acidity would still be 7.0 pH (or effective neutrality so far as inversion is concerned).

Blowski and Holven worked out a method of pH determination using the spot-test method against colored celluloid standards which is simple and effective. (See p. 280.) They also developed a system of regulating the reaction by the addition of lime at

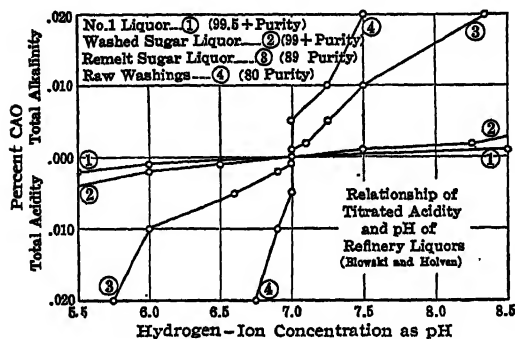


FIG. 49.—Titrated Acidity and pH of Refinery Liquors.

various points in the house instead of at the blowups only, which they report uses less lime and maintains the pH of all products at close at 7.0.

### CHAR FILTRATION

**167. Purpose and Importance.**—The liquors which have been clarified by defecation and cloth filtration, or other means, contain coloring matter and impurities which must be removed before refined sugar can be produced economically. The material used as a decolorant is animal charcoal or boneblack, usually called "black" or "char" (in some few refineries, "bone coal") which is the granular residue obtained by the destructive distillation of bones. Boneblack was first employed in sugar refining about 1811, being introduced into the liquors in powdered form and then strained out. Its development as a granular filtering medium dates from 1828. Although primarily used as a decolorizer it has the property of removing organic and inorganic impurities and this property is of the utmost value, since the removal of these impurities reduces the amount of residual sirup ("barrel sirup") or waste molasses, and increases the yield of refined sugar accordingly.

The economy and efficiency of the refining process depend upon careful attention to detail in the char house; exact regulation of the densities, temperatures and reactions of onflowing materials; constant attention to the color, speed of flow and brilliancy of the effluent liquors; control of the revivification and the physical condition of the boneblack itself and finally maintenance of a definite time schedule or "cycle" for the various steps of the filtering operation. The elaborate control is further complicated in large refineries by the fact that two or three grades of boneblack are maintained, each having its own



equipment of filters, kilns and handling apparatus. Many of the liquors are double and triple filtered, which adds to the complications of char-house procedure and control.

**168. Char Filters.**—The filters are cast-iron vertical cylindrical cisterns, conical at the top and bottom, and usually about 10 feet in diameter and 20 feet deep (1200 cubic feet capacity). The top is closed by a movable door termed the filter-head. Two manholes at the sides near the bottom serve for the removal of the exhausted black. The char rests on a perforated plate covered with a coarse-weave cotton blanket, and this in turn with a blanket of finer weave, to prevent the char-dust being carried out with the filtered liquor. The inlet-pipe is at the side of the filter, close to the top, and the outlet is at the bottom, below the perforated plate. The outlet-pipe is carried up in a gooseneck on the outside of the filter to within a few feet of the top. Figure 50 shows the general arrangement of a char-filter and its accessory equipment.

**169. "Char Capacity." Boneblack Ratio.**—The ratio of boneblack used to sugar melted varies widely in different refineries. The amount used depends on the available equipment, the quality of raws melted, the grades of refined turned out. A refinery making a relatively large quantity of soft sugars on a low-test melt would use 100 pounds of black or more per 100 pounds of raw melted. (This means that the same amount of boneblack will be revived daily as sugar is melted. The actual boneblack in process, including that in filters, driers, kilns, elevators, etc., will be about two and a half to three times the daily melt.) With the high grade raws now being received and the reduction in the demand for soft sugars, most refineries operate on a much lower boneblack ratio—50 to 60 pounds of black per 100-pound melt.

Modern refineries generally have from 24 to 36 char filters of the size described above, for each million pounds melted, and from one-third to one-half as many kilns as filters.

**170. Filter "Cycle." Schedule of Operations.**—Every filter goes through the successive steps of the cycle according to a time schedule, this schedule being based on the length of time that it takes to fill a single filter with char (which is in turn dependent on the rate that the kilns are delivering revived black) together with the number of filters operating in the cycle. Since each filter must be ready in turn to take its filling of "burned" black it is evident that the total time of all the operations of the filter cannot exceed the time of filling one filter multiplied by the number of filters.

It is customary in most char houses to separate the boneblack into two or three grades, designated by letters or numbers. Let us assume that a filter-house has 36 filters, 18 of which are used for the "A" grade boneblack and 18 for the "B" black. Considering only the first of these, the "A" filters, and assuming that the number of kilns allotted to that grade of black is sufficient to revivify a filter-full of black every four hours, the "cycle" of the "A" filters would quite evidently be seventy-two hours. That is, each of these filters must go through these successive operations in that time: (1) Filling with char, (2) Covering the char with liquor ("settling" the filter), (3) Running liquor, or the filtration proper, (4) Washing the sugar out of the char with

hot water, (5) Blowing out the residual water with air, (6) Discharging the wet char, or "dropping" the filter. Filter schedules vary through wide limits, 48 to 54 hours being the shortest and 90 to 100 hours the longest. All these operations except (3), Running liquor, are more or less fixed, no matter what the length of the cycle, so the variable is generally the time allotted to running liquor, *i.e.*, the filtration proper.

Two systems of working char-filters formerly prevailed: The "set" or "battery" and the "continuous" systems, but the "set" system has now been largely discontinued. In the set system, all the filters filled in one day were worked as a group unit, all the filters in the group doing the same class of work at the same time. In the continuous system, each filter goes through its cycle independently, a step ahead of the filter filled immediately after it. The set system was used by refineries making a large percentage of soft sugars because it lent itself more easily to their pan-boiling system and also simplified the use of double- and triple-filtration. The continuous system requires a much smaller char-filter installation for a given melt, so with the reduction in soft sugar demand it has superseded the set system. The handling of any one filter in the two systems is essentially the same.

**171. Filling the Filter with Char.**—The distribution of the boneblack in the filter is of primary importance. If the dust and larger grains of char are not evenly distributed throughout the filter, the liquor will flow through the coarser particles, forming channels and causing many difficulties during the washing-off period. Even distribution may be obtained in many ways and there are several patented devices designed to secure this result. The char may be delivered into the filter by means of a funnel with a slightly bent stem, the funnel being turned at intervals by hand or continuously by a motor. The most effective method of filling the filter with dry char is to deliver it into the filter promiscuously and have a man enter the cistern from time to time and distribute the material with a shovel. After the filter has received sufficient char, this is drawn up into a cone in the middle.

H. I. Knowles<sup>2</sup> in a study of boneblack filtration showed that even with hand-levelling ten or twelve times during the filling of a filter the distribution of char leaves much to be desired. The sides of the filter contained char of much coarser grist than the center, and samples taken later during the filtration period by special sample pipes inserted in the filter showed higher color, ash and organic removal in the center of the filter than at the sides. This was in large part due to more rapid flow and less contact through the coarse char of the sides than in the fine char in the center. "Wet filling," *i.e.*, adding the char and liquor to the filter together has recently been adopted to avoid this separation of coarse and fines and will be described later.

**172. "Covering" or "Settling" the Boneblack with Liquor.**—Air-pockets in the char must be avoided. To prevent these, with dry filling as described above, the liquor is delivered slowly at the base of the cone of char, with the head of the filter off. The liquor runs down the sides of the filter until it reaches the blanket, then flows across the whole width of the filter at the bottom and rises, forcing the air out through the cone of char, which is kept dry

<sup>2</sup> Ind. Eng. Chem. Vol. 17 (1925), No. 11.

throughout the covering. When the liquor begins to flow from the goose-neck the outlet valve is closed and liquor is run in until the filter is filled. In spite of all precautions there will always be some entrapped air and this is permitted to escape by allowing the filter to "boil" as filter-men call it. After there is no further evidence of escaping air the head is put on the filter and the liquor is turned into it under pressure (15-25 feet head). The covering usually requires about four hours.

Considerable heat is generated when hot liquor is first run on to well-burned char. With the char at a temperature of 140° to 150° and the liquor at 165°, the outflow during the first few hours of running will normally have a temperature of 185° to 190°. If covering is stopped under these conditions, or if filtration is suspended during the early stages, the temperature in the filter may rise to such a point that the liquor will be scorched. The char should be cooled to at most 160° before it enters the filters, and every precaution should be observed to avoid interruptions to the flow of liquor during the covering period and until the temperature of the outflowing liquor is practically that of the liquor entering the filter.

Knowles in the study already referred to showed that the temperatures of the liquor at the side of the filter are much lower than in the center where he found liquor rising to 210° F. during the first twelve hours of running.

Covering filters by bottom-filling has been advocated but has never found much favor in American refineries. In this practice liquor (generally once-filtered washed sugar liquor) is run into the bottom of the filter through the outlet pipe. As the liquor rises in the black the voids are completely filled and there is less opportunity for entrapped air or air-pockets to form. The practice might possibly have some advantages over top-filling and covering with dry char but is not as advantageous as the wet filling method described in the following paragraph.

**173. Filling and Covering Together. "Wet Filling."**—The process of intimately mixing the char and the liquor and adding them together to the filter has been tried out from time to time through many years but a workable procedure has only recently been developed. The method as now used was worked out in the Californian and Hawaiian Refinery and consists of first adding dry char until the filter is filled one-fifth to one-fourth of its volume and then running in the char and liquor together through a set of three eccentric funnels placed one above the other and hung in the mouth of the filter. The eccentric arrangement gives the char and liquor a swirling motion which mixes the two, the flow of char and liquor being so adjusted that the mixture falls from the bottom funnel at about the consistency of a loosely mixed concrete. With a little care this mixture can be so maintained that there is no tendency for the fines and coarse to separate. If the mixture is too free (*i.e.*, too great a proportion of liquor to char) this classification of coarse and fine will occur, whereas with too dense a mixture dry pockets of char will occur in the filter and give trouble later both in the filtration and washing periods. It has been found advantageous in one refinery to measure the onflowing liquor by means of a measuring box similar to that used at the liquor gallery for measuring outflowing liquors. The flow of char is very regular and by adjust-

ing the liquor flow by measurement all difficulties are avoided and close attention is not required.

In practice this wet filling method has reduced "channelling" of liquors to a minimum; has cut down the time of filters "in sweet water" about 25 per cent and has practically eliminated bad washing after sweet water. About three hours' time is also added to the time of filtration by wet-filling which is otherwise taken up in separate filling and covering. It is probably the greatest improvement in char filter technique of the past ten years. The first liquor which flows from the filter is generally cloudy but after twenty minutes or half an hour of running to refiltration this clears up and the liquor is brilliant.

174. The Filtration.—As soon as the "covering" is complete and the filter-head is in place the filtration proper begins. Practice in refineries differs as to how the filtration schedule is conducted, depending on the "char capacity" (boneblack ratio), the character of the melt, the number of grades of char, the amount of refiltration and the quantity of soft sugars to be made, besides many purely local considerations. Certain general principles are followed, however, in all refineries. The highest purity liquors are filtered first and followed by liquors of lower purities. Washed sugar liquor (99°) goes on the char at 160°–165° F. and the same brix that it comes from the cloth filters, viz., 57°–60°. The speed of flow is from 150 to 250 cubic feet per hour. The density of the lower purity liquors is usually somewhat less than those of highest purity and with each successive grade that is filtered the speed of flow is reduced and the temperature increased. Each grade of liquor follows the preceding one without a break in the filtration; the "swing-pipe" at the top of the filter inlet being shifted from the delivery line of one liquor pressure-tank to the other, after which the filtration proceeds.

Where two or three grades of black are maintained the principle is followed that the lowest black receives the materials having the largest amount of impurities, while the liquors for refiltration are filtered through the best grade of black. The reason for this is apparent since the impurities removed by the first filtration are in greater quantity but are the easiest to remove while those removed by refiltration are present in less amounts but require a more active boneblack for their removal. Furthermore, overworking the most active black would quickly reduce its efficiency.

Washed sugar liquor forms the bulk of the liquor to be filtered so it is generally run on each filter for 20 to 24 hours. The effluent is water white and above 99° purity and goes to the vacuum pans for boiling to granulated sugar. After many hours running the No. 1 liquor assumes a yellowish tinge and it is then used for covering other filters, or may be refiltered. Following the washed sugar liquor, remelt sugar liquors of 94 to 97 purity may be filtered, then "granulated sirups" (90° purity) which are sirups from granulated sugar strikes too dark for further boiling to granulated. Cloth-filtered raw sugar washings (affination sirup) of about 80° purity and sirups from remelt sugar strikes are the low-grade materials which are generally sent to the char-filters. These and the granulated sirups are double and triple filtered to make light-colored liquors of low test for soft sugar manufacture or for fancy sirup

production. The second and third filtrations remove color but do not materially raise the purity of the liquors.

Some refineries of limited char capacity do not filter anything of lower test than granulated sirups of 90° purity. They are necessarily limited in the extent to which they can turn out soft sugars. Where such practice is followed the raw sugar washings are sent to the vacuum pans direct without cloth- or char-filtration and are boiled to remelt sugars.

**175. Washing Off.**—At the end of the filtration period the boneblack, so far as regards color removal and adsorption of impurities, is practically exhausted. The inlet-valve is now closed and the level of the liquor in the filter is lowered by gravity or air-pressure to that of the char. The head of the filter is removed and the char is leveled and then hot water (210° F.) is run upon it until the filter is filled. The head is replaced and hot water is run into the filter under pressure. The rate of flow of the water is about half that used for the liquor. The idea is to have the water displace the liquor remaining in the filter with as little mixing of the two as possible. This is so well accomplished in a well-running filter that there is no appreciable break in the density of the outflowing liquor for six or eight hours after water is put on the char. When the dilute liquor has a density of about 40° Brix it is termed "char sweet water," and the rate of flow is now further reduced and the filtrate is sent to the multiple-effect evaporator for concentration with other sweet-waters. The density and purity of the sweet-water fall rapidly. After three to five hours the water no longer contains enough sugar and is of too low purity to warrant its evaporation and it is turned to waste.

The char sweet-waters contain very high percentages of ash on the solids present and they also are high in colloidal material.<sup>4</sup>

The ash and colloids which have been adsorbed during the filtration are "leached out" of the black in the dilute runnings after water has been put on. It is obviously the best practice to keep the evaporated char-sweet waters separate from other materials containing less ash and colloidal impurities. They should not be returned to the boneblack for refiltration and the use of char sweet-water for melting purposes should never be considered.

**Washing to Waste.**—The washing to waste is continued for at least ten to twelve hours, the speed of flow being again increased to about 200 cubic feet per hour. Washing for a longer period will give more effective results and is the practice when time admits. Much of the organic matter is so strongly held by the char that no amount of washing will remove it. At the completion of the washing-period the water is driven out of the char by compressed-air, 10 pounds pressure for two hours usually being sufficient; the bottom doors of the filter are opened and the char is discharged into the hoppers on top of the driers. (Fig. 50.)

**176. Revivification of the Boneblack.**—The revivification of the black is accomplished by heating it to 1000° F. or higher out of access with the air. Special driers and retort-kilns are designed for this purpose. The "burning" of the black, as it is usually called, distills off some of the organic impurities

<sup>4</sup> Paine & Badollet. The Planter, Vol. 79 (1927), No. 2.

which the washing failed to remove, and carbonizes the remaining portion.

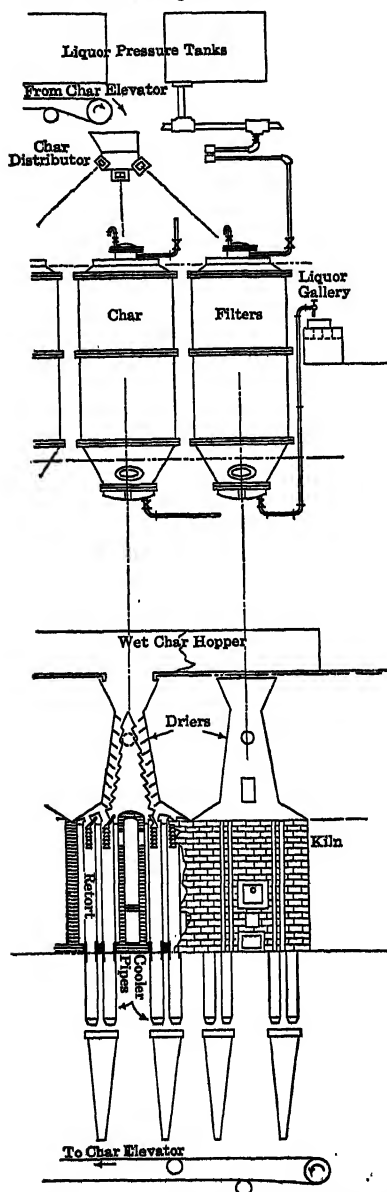


FIG. 50.—Char Filters, Driers and Kilns.

The alteration of boneblack with use will be discussed later.

*Char Driers.*—The wet char is passed by gravity through driers preliminary to the revivification. The driers are of various designs and in general consist essentially of a casing through which the waste gases from the kilns are drawn. Deflecting plates conduct the wet char slowly over the surface of this casing. The char enters the driers carrying about 18 to 20 per cent of moisture and leaves them with about 12 to 15 per cent.

*Char Kilns.*—The kiln consists of a furnace flanked on both sides by double or triple rows of upright pipe retorts, 20 retorts to the row, through which the char slowly passes. The commoner type is the double-row, or 80-retort, kiln. The retorts are of cast iron, of oval cross-section, 3 inches by 9 or 3 inches by 12, from 8 to 10 feet long. The combustion chamber occupies about one-quarter the width of the kiln, running through the entire length, and is fired at both ends, the fuel being coal, oil or natural gas.

The products of combustion leave the chamber near the top, are conducted downward along the length of the retort to ports at the bottom of the kiln, and thence by ducts in the brickwork up to the casing of the char-driers. Recording pyrometers are now general for regulating the temperature, the practice in most refineries being to place the thermocouple between the rows of retorts in the center of the kiln at the hottest spot. Temperatures between 1000° F. and 1200° F. are usual. Peep-holes in the brickwork permit of judging the

temperature of the retorts in the absence of a pyrometer, a dull red heat being necessary for ordinary revivification. At the base of each retort is fitted a sheet iron cooler-pipe identical in shape with the retort and extending below the kiln some 6 or 8 feet. The cooler-pipe takes the hot char at the kiln temperature and cools it to the point where it can be admitted to the atmosphere without danger of the carbon burning. The speed of the char through the kilns is controlled by means of a cam arrangement actuating a small draw-plate or tilt-bucket at the end of each cooler pipe.

Each 80-retort kiln will revivify about one filter (1200 cubic foot) of char per twenty-four-hour day, or about 15 cubic feet per retort per day. The char takes about five hours to travel through the retort. Kilns may be forced to 50 per cent greater capacity than this but only at the expense of the furnace linings and retorts. From the cooler pipes the char falls into hoppers that deliver it to the conveying system (endless belts and bucket elevators) by which it is carried to the distributing system at the filter-heads where it is run into an empty filter to go through the cycle again. (See Fig. 50.)

The char should never go to the filters at a temperature above 160° F. (many refiners prefer even lower temperatures) so that it is generally necessary to further cool the black on its way to the filters by passing it over pipes through which cold water is circulated. A simple method of cooling the char is to spray it with water in a fine spray as it passes along the conveyor belt beneath the kilns. The water all evaporates in the passage of the char through the conveyor system, the cooling being very effective and easily regulated.

*Weinrich Decarbonizer.*—The reburning of the black carbonizes some of the organic matter which the washing failed to remove. This carbon remains in the pores of the black, and since it has no decolorizing power such as is possessed by the constituent carbon, it merely clogs the pores of the char and decreases its filtering value proportionately. The Weinrich decarbonizer, designed to remove this added carbon, is a revolving drum, slightly inclined to the horizontal, with a carefully regulated fire under it. The vegetable carbon is burned away as the char passes through the drum, thus increasing the porosity and prolonging the life of the boneblack.

*Renewal of Boneblack.*—Mechanical handling causes the grain of the boneblack to be broken down by attrition. The fine dust is removed by means of suction fans which draw air through the char as it falls over umbrella-shaped devices, generally placed at the discharge of the elevators just ahead of the char-distributing system to the filters. Besides this dust removal it is occasionally necessary to screen out the finest char (below 50 mesh) with wire-mesh screens, the mechanical shaking type being employed. The dust was formerly used in making phosphoric acid for the defecation but is now sold for fertilizer manufacture and other purposes. The char also decreases in bulk somewhat by shrinkage in the burning.

These losses are made up by the addition of new bone-black, generally a filterful at a time. In refineries having two or more grades of black, the new black is naturally added to the highest grade, and the heaviest screening is done on the lowest grade. As new black is added to the first grade, an equal volume is thrown from the first to the second and from the second to the third

to maintain the proper proportions. As will be explained later, new black must be washed and burned before use. From 15 to 30 per cent of new boneblack is added yearly, depending on char capacity and other factors. The boneblack in a refinery is therefore completely renewed every three to six years.

177. *Composition of Boneblack and its Alteration by Use.*—Analyses of new boneblack of American manufacture and of the same black after several months of use, are here given:

	New Black	After Six Months' Use
Carbon.....	8.55	10.30
Insoluble silica.....	.15	.31
Calcium sulphate.....	.06	.97
Calcium sulphide.....	.05	.24
Calcium carbonate.....	8.90	4.20
Iron.....	.06	.23
Undetermined (calcium phosphate).....	82.23	83.75
	100	100.00
Weight per cubic foot, loose.....	43.8 lbs.	54.5 lbs.
Weight per cubic foot, packed.....	48.5	59.1
Percentage between 16 and 30 mesh sieves.....	84.8	66.1

Boneblack always contains some nitrogen, but the rôle this plays in the filtration has not been determined. It has been long believed to be essential to the decolorizing power and Patterson<sup>5</sup> and later Hall<sup>6</sup> extracted a nitrogenous body from boneblack by digestion with sulphuric acid, a few drops of which were claimed to have the decolorizing power of several grams of black. When precipitated on inactive wood charcoal this material also was supposed to give a highly active carbon but these results were explained shortly after by Tanner<sup>7</sup> as being due to the decolorizing action of the sulphuric acid on the caramel used in the tests and not to the nitrogenous body. Hauge and Willaman<sup>8</sup> failed to find any decolorizing power in the nitrogenous body so extracted, and other investigators have presented arguments against the decolorizing power of the nitrogenous constituents of the char which seem to be conclusive. New black contains a considerable amount of ammonia salts, which must be removed, since they would have a detrimental effect upon the color of the

<sup>5</sup> J. Soc. Chem. Ind., Vol. 28 (1909), 700.

<sup>6</sup> The Planter. Vol. 68 (1922), No. 2.

<sup>7</sup> Ind. Eng. Chem., Vol. 14 (1922), p. 441.

<sup>8</sup> Ind. Eng. Chem., Vol. 19 (1927), p. 943.



filtered liquors. For this reason, new char must be thoroughly washed and burned before use for filtration.

As is shown by the analyses, the composition of char alters with use. The carbon increases steadily because of the deposition of inert carbon in the pores during the revivification. The calcium sulphate increases by the removal of sulphates from the liquors and the water. The sulphide tends to increase as the sulphates increase, due to the reduction of the latter by the organic matter during the burning. The calcium carbonate drops sharply during the first few washings and burnings and finally tends to reach a balance around 4 per cent. The iron increases slowly. Of all these impurities the calcium sulphide and iron are the most objectionable. Since appreciable quantities of these two constituents will give a greenish color to the filtered liquors and a gray cast to the sugars boiled from them. A boneblack may become unfit for use if the calcium sulphide content exceeds 0.4 per cent. Thorough washing and burning, together with the constant addition of new black, will prevent the sulphide content from becoming abnormal.

The weight per cubic foot tends to increase due to two reasons: the bulk and size of the grains shrink with use and the pores of the black become clogged with adsorbed impurities and inert carbon. Since the action of char is due to adsorption, which is a surface phenomenon, the less porosity, the less adsorption. The weight per cubic foot is therefore, in a sense, a measure of the activity of the char.

**178. Action of Boneblack on Sugar Solutions.**—Boneblack is primarily a decolorant but its ability to remove organic and inorganic impurities adds enormously to its value to the refiner. It is now generally agreed that it acts by adsorption, following Freundlich's equation which is, that for any given temperature  $X/M = KC^{1/n}$  in which  $X$  = amount of dissolved substance adsorbed by  $M$  grams of char;  $C$  is the concentration of the dissolved substance at equilibrium (*i.e.*, after the adsorptive action has ceased), and  $K$  and  $1/n$  are constants depending on the amount and kind of the solution treated with the char. Within the working limits for sugar solutions the higher the temperature the greater the adsorption. Obviously, the upper limit for sugar work is about 180° to 185° above which point inversion and decomposition become possible factors.

**Color Adsorption.**—M. T. Sanders<sup>9</sup> and F. W. Zerban<sup>10</sup> showed that the adsorption formula as given above is valid for color removal by all carbons including boneblack when the "color units" of Meade and Harris<sup>10a</sup> are substituted for the concentrations  $X$  and  $C$ . (Other color units based on the extinction coefficient (264) may also be used.) It should be noted that adsorption isotherms when plotted for  $X/M$  and  $C$  on logarithmic cross-section paper are straight lines.

The claim that the decolorizing power of boneblack rests in the nitrogen content has been pretty well refuted (see p. 170) and the carbon deposited on the bony structure of the black at the time of manufacture is now recognized

<sup>9</sup> The Planter, Vol. 71 (1923), No. 6.

<sup>10</sup> Unpublished, presented before Am. Chem. Soc., 1921.

<sup>10a</sup> Ind. Eng. Chem. Vol. 12 (1920), p. 687.

as the color adsorbing medium. Refiners have long known that the adventitious carbon deposited in the pores during revivification not only has no decolorizing value but is actually a detriment in that it reduces the porosity of the black and in consequence its activity. H. I. Knowles<sup>11</sup> suggested that there is an optimum carbon content giving the highest decolorization and in experiments in which he decarbonized and recarbonized chars in the laboratory found the optimum decolorizing value at a carbon content of about 6 per cent. Some boneblacks which were in refinery service were studied by him during twenty months and found to be at their optimum decolorizing value when the carbon had reached 7 per cent. It seems probable that this figure would vary considerably for different chars, and that other conditions might have a greater effect on the decolorizing power (*e.g.*, the alkalinity of the black and the extent of the revivification).

The pH of the solution going on the black is of great importance as was shown by Hauge and Willaman<sup>12</sup> in a study which ascribed to the disregard of this feature most of the discrepancies and contradictions in the work of different investigators on decolorization by vegetable carbons. They showed that caramel is readily adsorbed by boneblack only at low pH values; *i.e.*, much higher acidities than are encountered in practical sugar work. Experiments such as these on pure caramel have probably been the source of the long-expressed belief that the reddish and brown coloring matters in raw sugars are hardest to remove by boneblack. Wayne<sup>13</sup> in a careful investigation of decolorizing efficiency by spectro-photometric analysis showed that no such generalization concerning caramel-types of coloring matter can be drawn. The colors due to the poly-phenols of iron (grayish-green and greenish-brown tints in washed sugar liquors) are more easily removed so far as percentage of color removal is concerned than the red coloring matters, so long as complete or nearly complete decolorization is not attempted. When the decolorization is carried to the extent that the refiner must carry it to give him liquors suitable for refined sugars, a residual color is encountered in the gray-green sugars that is highly resistant to char adsorption and the resulting sugars have a grayish cast, whereas the red coloring matter from incipient caramelization may be completely removed, yielding white liquors free of a gray cast.

Char-filtration removes about 80 to 90 per cent of all the color entering the refinery in the raw sugars. The high-grade liquors (washed sugar liquor 99 purity) are decolorized practically 100 per cent.

*Ash Adsorption.*—The removal of inorganic matter is due in general to the bony structure of the char, as may be proved by burning off the carbon, when the residue will give as great, or sometimes greater, ash removal than before ignition. The selective action of boneblack for various kinds of salts was brought out a great many years ago; the phosphates, sulphates and carbonates being readily adsorbed while the chlorides and nitrates are adsorbed to a much lesser extent. Knowles<sup>14</sup> corroborated these facts as did Rice and

<sup>11</sup> Ind. Chem. Eng. Vol. 19 (1927), No. 2.

<sup>12</sup> *Ibid.* Vol. 19 (1927), No. 8.

<sup>13</sup> *Ibid.* Vol. 18 (1926), No. 8.

<sup>14</sup> *Loc. cit.* Vol. 19 (1927), p. 222.

Murray<sup>15</sup> and they showed as well that the nature of the base was of equal importance. Iron and calcium are more readily adsorbed than sodium and potassium. Similar selective action is evidenced for the organic salts. A considerable portion of the ash that is adsorbed by the boneblack during filtration is released to the thinner runnings and sweet waters after water is put on. This "leaching out" of the ash makes the char sweet waters very high in ash-sucrose ratio and their return to the char-filters after concentration should be avoided.

An important investigation on the action of boneblack by T. B. Wayne<sup>16</sup> deals largely with ash adsorption. His findings may be summed up as follows: (1) The percentage of ash adsorbed by a char increases regularly with increasing alkalinity of the char itself up to a pH of 9.6 as measured on a distilled-water extract of the char. (2) The pH of the boneblack is directly affected by the temperature of revivification; *e.g.*, a char burned to 900° F. (480° C.) (as measured by a pyrometer in the kiln) gave a water extract pH of 7.0, the same char burned to 1000° F. (538° C.) gave 8.4 pH, and when this char was burned to 1150° F. (620° C.) the pH was 9.3. From conclusions (1) and (2) it follows that ash adsorption is increased by increasing the temperature of the revivification. (4) The pH of the filtered liquor is increased as the pH of the char increases. (5) Varying the proportion of char to liquor does not alter the pH of the filtrate. This in conjunction with (4) shows that the alkalinity of the black is held in an adsorbed state and is liberated to the liquor in exchange for some of the constituents of the liquor until an adsorption equilibrium is reached. (7) The caustic soda test (see 189) may be "colorless," *i.e.*, excellent revivification may be indicated, but if the pH of the black is not high enough due to too low a temperature of revivification, acid liquors may result, even though both the black and the on-flowing liquors may be above 7.0 pH.

This last finding is demonstrated by Wayne by filtering neutral calcium acetate solution (pH 7.3) through a char burned at various temperatures. The results are shown as follows:

pH of Char Extract	pH of Calcium Acetate Solution On	pH of Calcium Acetate Solution Off
6.9	7.3	6.0
8.2	7.3	7.0
9.6	7.3	8.4

It has long been recognized by some refiners that poorly burned boneblack in refinery service yields acid liquors and increases inversion losses. This was ascribed to various causes, but the above findings show it to be due to a

<sup>15</sup> Ind. Eng. Chem. Vol. 19 (1927), p. 214.

<sup>16</sup> *Ibid.* Vol. 20 (1928), No. 9.

form of "hydrolytic adsorption" *i.e.*, a breaking up of neutral salts into acids or acid salts within the black.

Wayne's conclusions in this investigation bear out many points which the writer has observed in actual char filter practice but which have lacked scientific corroboration until now. Principal among these are the need of relatively high temperatures and exact control in revivification, the fallaciousness of the idea of "over-burning," and the fact that the pH of the filtered liquors is not dependent on the pH of the on-flowing liquor but on the condition of the boneblack itself.

From 20 per cent to 50 per cent of the total ash entering the refinery will be adsorbed by the boneblack. Many factors will influence the variations between these limits as may be judged from the previous discussion.

*Adsorption of Invert Sugar.*—There is no question that the reducing sugars in the char-filtered liquors are lower than before filtration particularly during the earlier runnings, but whether this is a selective adsorption or a destruction due to the alkalinity of the black is a doubtful point. Another point in question is whether the "glucose" is leached out after water is put on as in the case of the adsorbed ash. In the last edition of this book the present writer (G. P. M.) stated that this leaching out did occur, the conclusion being reached from observations of filters in practice. Blake<sup>17</sup> concluded from similar observations that levulose is selectively adsorbed in excess of dextrose<sup>18</sup> but that later this was given up to the sweet-waters. An extended attempt to prove this point by laboratory experiment<sup>19</sup> has failed to show a single instance of such leaching out after the disappearance of the invert sugar (or "glucose") nor have any char-filters been found in house practice since pH control was developed where the glucose ratio in the sweet-waters is higher than in the highest glucose-containing material going on the black, provided all materials including sweet-waters are above 7.0 pH. It appears that the apparent leaching out occurs only when acid conditions occur in the sweet-waters or thin liquors, which would lead to the conclusion that the increase in glucose observed by some investigators is due to an inversion and not a leaching out. However, it may also be that the alkaline substances formed in the char during revivification aid in the adsorption of the glucose as they do in the ash adsorption as shown by Wayne, and that when this alkalinity disappears the glucose is released to the sweet-waters.

The matter is of practical importance to the refiner since it involves the question whether the refinery should show a "glucose balance" or a "glucose removal" for its entire operations, if no inversion or destruction of glucose is taking place elsewhere.

*Adsorption of Colloids.*—Much of the coloring matter of sugar solutions is colloidal but there are also other colloidal non-sugars present. Paine and Badollet<sup>20</sup> studied colloid elimination during char filtration and found that the

<sup>17</sup> The Planter. Vol. 65 (1920), No. 22.

<sup>18</sup> This selective adsorption of levulose from invert sugar is reported by Nakamovitch and Zelikman; Facts About Sugar (Abstract), December 3, 1927.

<sup>19</sup> Meade and Baus. Presented at Philadelphia A. C. S. Meeting, 1926.

<sup>20</sup> Facts About Sugar. Vol. 22 (1927), No. 3.

"irreversible" type (high in ash; do not readily redisperse when dried and taken up in water again) are preferentially and completely removed during char filtration while "reversible" colloids (gummy materials) are never completely adsorbed and are leached out in the sweet-waters.

**179. Refining with Vegetable Carbons.—*Suchar Process.*** Refining with activated carbons instead of boneblack is fairly general in Europe but only one variety—"Suchar"—has been developed to a commercially successful process on this side of the water. There are small refineries in Porto Rico, Santo Domingo, Louisiana and elsewhere using suchar only and producing refined sugar. The process is briefly described by the Suchar manufacturers as follows:

The raw sugar is washed up to 98.5° to 99° and melted. The melt is then subjected to a double filtration with Suchar according to a special procedure known as the "Multiple Density Process," which enables the refiner to send to his pans a highly purified liquor having a density of from 62° to 64° Brix. The quantity of carbon used is very small, averaging from 1 per cent to 1½ per cent on the solids in the melt, and the filtration takes place in automatic filters of special design.

The exhausted carbon is sweetened off, dried in a continuous dryer and then re-activated in a continuous electrical furnace which is patented for the Suchar Process. The carbon is discharged from this furnace in a cold, dustless condition in which form it can be conveyed back to the mixing tanks for treating a further quantity of melt.

**180. Comparison of Boneblack and Decolorizing Carbons for Refining.—**Blowski and Bon<sup>21</sup> made a comparative study of carbons and boneblack for refining purposes and gave the following advantages and disadvantages of the two processes:

*Boneblack. Advantages.*

(1) Elimination of Non-sugars. Approximately 1 per cent of non-sugars on melt is eliminated, resulting in a decreased molasses production and an equivalent increased sucrose recovery.

(2) Ease of Regeneration. Spent bone-char may be readily restored to practically its original efficiency by a process of washing and kilning.

(3) Small Replacements. The replacement of char due to shrinkage and dust removal is small—in a carefully managed plant amounting to between 13 and 15 per cent per annum of the char in process.

(4) Highly Developed Technology. Process is already highly developed.

*Boneblack. Disadvantages.*

(1) Large Investment in Plant. Char equipment and the large amount of char in process necessitate a large investment.

(2) Large Amount of Material to be Handled. There is a great bulk of bone-char and liquor in process at all times.

(3) Large Wash-water Consumption. A large quantity of hot water is necessary for the washing.

<sup>21</sup> Ind. Eng. Chem. Vol. 18 (1926), No. 1.

*Decolorizing Carbons. Advantages.*

(1) **Simplicity of Process.** Because of high decolorizing power and rapid action, the amount of carbon required to decolorize a given quantity of liquor is small, and the time in process is short.

(2) **Reduced Inversion Losses.** Owing to the shorter time that sugar liquors are in process, there should be smaller inversion losses than exist with the bone-char process.

(3) **Smaller Investment Necessary.** No large quantity in process and equipment is simpler.

(4) **Small Wash-water Requirements.** The washing requires no such volume of hot water as does the washing of bone-char.

*Decolorizing Carbons. Disadvantages.*

(1) **Low Non-sugar Elimination.** The non-sugar elimination is for all practical purposes negligible, because amount of carbon is very small and only high-purity liquors are treated.

(2) **High Losses in Regeneration.** Losses will amount to at least 5 per cent per cycle. With the price of carbon over \$300 per ton the replacement cost is no small item in the cost of manufacture.

(3) **Increased Remelt Boiling.** Because of negligible elimination of non-sugars calculations show that the burden on the remelt pans would be increased by at least 140 per cent.

*Carbons and Boneblack in Conjunction.*—This combination has not been tried in this country but it offers some possibilities that warrant serious consideration. Dedek and Kael<sup>22</sup> found that carbon acting before boneblack (never in the reverse order) increases the ash adsorption because the active carbon splits up the organic salts of sodium and potassium by hydrolytic adsorption, and adsorbs the liberated anions, while the boneblack adsorbs the kations. The work of Paine and Badollet on adsorption of colloids by boneblack (see p. 174), shows that boneblack adsorbs irreversible colloids much more readily than the reversible type (gums, etc.), while it is well known that carbons adsorb the reversible colloids most easily, another evidence of complementary action of the two types of decolorants. The selective removal of color by the carbon and by boneblack is also different which is another argument for their inter-action.

## CRYSTALLIZATION OF THE SUGAR

**181. Classification of the Liquors.**—The liquors are classified at the filter outlet-pipes according to their purity and color and are distributed to storage-tanks near the vacuum-pans. In the continuous system of filtration, in which no two filters are at the same phase of their cycles, all grades of liquors are flowing at the same time. The liquors are classified about as follows: First liquor: Filtered washed-sugar liquor, "water white" and of 99° to 99.5° purity; second liquor: Filtered granulated-sirups, off-white or slightly yellow and of

<sup>22</sup> Facts About Sugar. (Abstract), July 30, 1927.

90° to 93° purity; third liquor: Filtered or double-filtered washings or low-grade meltings, golden yellow and of 84° to 87° purity; fourth liquor: Last runnings, too dark to make granulated sugar and of 75 to 80° purity. As has been stated above triple-filtration of these liquors for soft-sugar work is common practice.

**182. Boiling to Grain.**—The vacuum-pans used in refining do not differ materially from those of the raw-sugar factory. The white sugar pans are usually of a lesser height in relation to their diameter so as to obtain greater heating surface per volume and to speed up the boiling. Twelve, 14 and even 16-foot diameter pans are employed. Calandria pans are now used for remelt sugars. Most refineries now evaporate in multiple-effect a part of the No. 1 liquor coming from the char filters, generally to a brix of 67° or even 70°. This heavy liquor is used to charge the pans thereby economizing both in time and in fuel. White sugar strikes using this heavy liquor are frequently boiled in as short a time as one hour. In general the time is from an hour and half to two hours.

The general principle of the manipulations is the same in the two branches of the industry, but from the nature of the product, greater care must be exercised in the refinery. This is due to the necessity of boiling sugars to certain specifications as regards the size and hardness of the crystals. The raw-sugar factory usually aims only to make sugars of a uniform size and hardness of grain. In boiling granulated and other hard sugars, a high pan-temperature is maintained (160°–180° F.), while for soft sugars, in which a small spongy grain is desired, low temperatures (120°–130°) prevail.

Selected water-white first liquors are used for the fancy grades of refined: cubes, cut-loaf, confectioner's sugar, etc. The bulk of the output is the granulated sugar of commerce, called in the trade "standard fine granulated" and the boiling system is planned for the production of this grade. The system generally employed is to have one or more pans continually working on "straight liquor" strikes, while the sirups purged from two of these strikes are combined and boiled in another pan to "first sirup strikes." These strikes yield still lower purity sirups which are combined and boiled to "second sirup strikes." The sirups purged from these "second sirup strikes" combine to make a strike which yields a sirup of about 90° purity and this is sent back to the char-house for refiltration where it is called "granulated sirup" or "sirup for boneblack," and returns to the pan floor as "second liquor."

The second liquor and the third liquor are boiled to make "off-granulated" sugar. This is a slightly "off-color" sugar which is disposed of by gradually and slowly mixing it with the better grade of granulated strikes.

The sirups from the off-granulated strikes are boiled with fourth liquor for "high remelt" strikes of 75° to 80° purity. The sugars of these strikes are washed to a high test (98° purity) and are melted directly with the washed raw sugar. The sirups from these strikes are boiled back on a footing of fourth liquor to make strikes of about 67° purity. These magmas are discharged into crystallizers where they are kept in motion during two to three days. They are then purged without the use of wash-water and yield a sugar from 85° to 90° purity. This sugar may be mixed with high-test raws and washed

with them, or, it may be melted directly with other low-grade raws and be submitted to the processes already described. Preferably, the sugar is mingled with a heavy sirup of low test, repurged and washed to give a 96°-98° test sugar which is then melted and sent to the defecators with the "high remelt" sugars.

The sirups purged from these low-grade remelt strikes form the residual sirup or "barrel sirup" of the refiners.

In the days when soft sugars were produced by some refineries to the extent of 25 to 30 per cent of the total output the pan-boiling system of the "soft-sugar houses" revolved around the production of softs, although the high-test liquors were boiled to granulated sugar in much the same way as that described above. Now few American refineries produce soft sugars beyond 5 per cent of their output<sup>23</sup> and the majority do not reach that figure so the boiling of soft sugars, though still a very important and profitable part of refining, has been more or less subordinated to the production of granulated.

Soft sugars are graded according to color, varying from an almost pure white to a deep brown sugar. The desirable characteristics from the trade point of view are texture, which should be spongy and free from any sharp crystalline appearance; color, which besides conforming to the desired grade should be free from grayish or greenish tones; flavor, freedom from harsh or salty taste; while the refiner seeks in addition to these keeping quality and low polarization. Soft sugars to attain these characteristics must be boiled from liquors free of turbidity, low in total color and free from greenish-gray cast, relatively low purity, low in ash and high in invert sugar content. These characteristics of the liquors are in part due to the character of the raws being melted but are largely dependent on char-filtration. Given the grade of softs which the market requires, liquors conforming to the characteristics and proper test are selected for the production.

The sirups from soft-sugar strikes are either boiled back directly or are first char-filtered, as the needs require. The varying demand for the different grades necessarily precludes the possibility of a fixed boiling system.

**183. Drying and Finishing the Product.**—The separation of the crystals from the sirup in the magmas is effected by centrifugal machines such as are used in the factories. The white sugars are very thoroughly washed in the machine to remove all adhering sirup. The moist sugars are conveyed from the centrifugals to distributing-bins above the granulators by belt conveyors or scrolls, and bucket elevators.

The bulk of the white sugar is sent through the granulators for drying. The granulator also separates the crystals from one another, hence its name.

The granulator is an iron drum (Fig. 51) about 6 feet in diameter and 25 feet long, slightly inclined to the horizontal toward the discharge end, and revolving on trunnions. A series of narrow shelves attached to the inside of the drum, longitudinally, serves to lift the sugar and let it fall through heated air as the drum revolves. The air is heated either by means of a steam drum extending through the middle of the granulator or by a large group of steam-

<sup>23</sup> The total production of soft sugars in the United States in 1927 was 3.7 per cent of the raws melted.



pipes at the discharge end of the drum. The air is drawn over the pipes and through the granulator by an exhaust fan which removes the sugar-dust. The illustration (Fig. 51) is of a Hersey granulator, the usual American type.

Two granulator-drums are usually operated in series, one above the other. The sugar leaves the upper drum partly dried and quite hot, hence very little steam is required to complete the drying in the lower section. The sugar should leave the lower drum comparatively cool, preferably below  $110^{\circ}$ . A third drum, connected in series with the other two, and containing no heating element, is sometimes used to cool the sugar. (See 138 concerning caking of sugars.) The sugar falls from the granulator to a set of screens which classify it according to the size of the crystals, remove the lumps and coarser dust, and deliver the products into the packing-bins. The screens formerly used were of the "bolter" type, being a revolving horizontal casing, inclined slightly, covered with wire screen of the desired mesh through which the sugar falls, the "tailings" being delivered at the far end of the bolter. The more modern

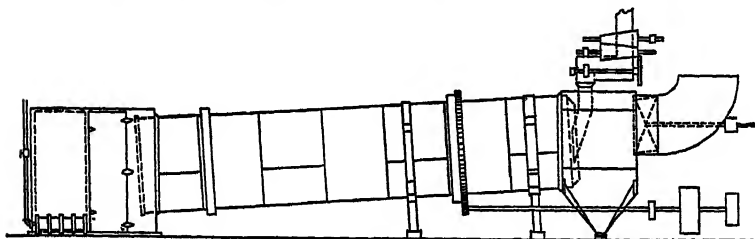


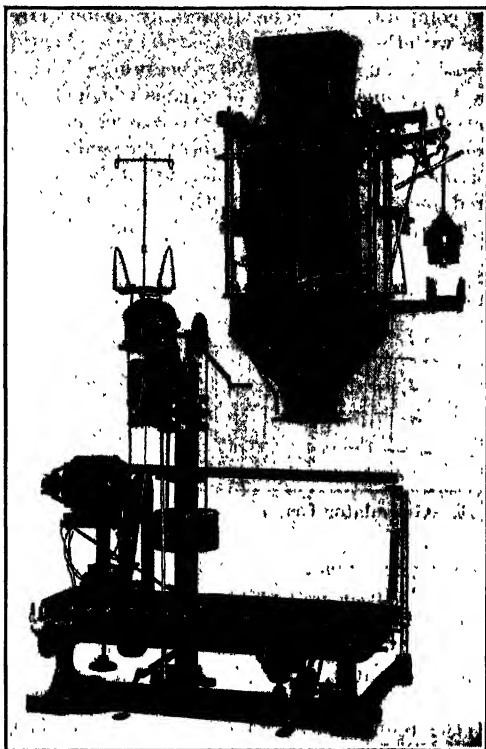
FIG. 51.—Granulator Drum.

screening devices are of the vibrating or tapping types. These consist of flat screening frames, inclined at about  $35^{\circ}$  to the horizontal, down which the sugar flows while mechanical devices tap or vibrate the screening surface to cause the crystals to go through the meshes. The screened sugar is packed direct as it comes from the screens, or may be sent to storage bins for packing during the daytime only.

The greater proportion of the refined sugar goes into 100-pound bags (either cotton-lined burlap or coarse heavy cotton) into which the sugar is automatically weighed and then sewed (see Fig. 52). Barrels, which formerly were in greatest demand, are now packed in relatively small proportions. Small cotton packages, paper packages, and cartons are all automatically filled, weighed and closed, the multiplicity of grades and packages making the "finishing house" of a large refinery an elaborate establishment in itself.

Granulated sugar now forms a large part of the output of all refineries and the total product of the smaller establishments. This grade is classified according to the size of the crystals as extra coarse, coarse, fine and extra fine granulated. The ordinary commercial granulated is the fine grade. Fruit granulated is the finer screenings from fine-grained sugar. As has been explained in an early chapter of this work, the size of the grain is determined in the pan-boiling.

**184. Other Classes of Refined Sugar.**—The various kinds of lump and loaf sugars are made by mixing sugar of the proper grain with a heavy white sugar sirup to form a moist mass. This is then variously treated by pressing or moulding to form the cubes, or slabs, after which the cubes or slabs are dried in hot-closets. The cubes are ready for packing after they have been dried.



*Hoegner Scale Co.*

**FIG. 52.—Automatic Scale.**

The slabs and bars are cut or sawed to form cut and sawed loaf-sugar. These are broken down in making crushed loaf-sugar.

Powdered or pulverized sugars of various degrees of fineness are made by grinding coarse granulated in a mill and passing it through bolting-cloth.

Brilliant or candy "A" is a large grain sugar which is barreled while moist, just as it leaves the centrifugal machine. Confectioners' "A" is a smaller grain sugar packed in the same way.

The soft sugars, white and yellow, have already been described. These sugars are packed while moist, directly from the centrifugals. The soft sugars are classified according to a series of arbitrary trade color-standards numbered from 1 to 16 and put out by the Sugar Institute, Inc. These standards are now

accurately graded by spectro-photometric analysis (see page 290).

**185. "Barrel Sirup."**—The sirups purged from low-grade remelt strikes form the residual sirup which is stored in large tanks as in the raw factory and then packed in barrels for the market or shipped in tank cars. As a rule the sirup is diluted to 55° Brix and reboiled "smooth" before barreling. Certain refineries pay especial attention to this sirup and char-filter it to improve the color before reboiling. Much of it is sold as "black-strap" in the form that it comes from the centrifugals.

This sirup varies in composition through rather wide limits, depending on the class of sugar from which it is derived and upon other factors. A typical

analysis would be as follows: Brix, 84°; polarization, 34 per cent; reducing sugars, 22 per cent; ash, 6 per cent; water, 22 per cent; organic non-sugar, 16 per cent.

**186. Yield of Refined Sugar.**—The yield of sugar in a refinery is expressed in percentage terms of the raw sugar melted. The following figures are illustrative: Polarization of the melt, 96°; granulated yield, 93.6 per cent; sirup yield, 5 per cent at 34° polarization (1.7 per cent of sucrose per cent of melt). Sucrose lost in manufacture, 0.7 per cent. If other sugars than granulated are made they are calculated to a basis of granulated sugar and sirup.

#### REFINERY TECHNICAL AND CHEMICAL CONTROL

**187. Introductory.**—The technical control of a refinery is more positive and direct than that of a raw-sugar factory because the weighing and analysis of the raw material entering the refinery offer no difficulty. All the control figures are computed upon the basis of the weight of raws. A technical statement may be made up with comparatively few figures—the analysis and weight of the raw sugar melted; the analyses and weights of the refined sugars and sirup produced; the stock-in-process figures for the beginning and end of the period.

*Technical Control.*—Each package of raw sugar is weighed and sampled as it is dumped and the samples are polarized at convenient intervals, usually twice daily. Complete analyses are made weekly, or as often as desired, of a weighted sample made up from the daily melts. Besides the average polarization, which is computed from the component polarizations, the invert sugar, moisture, ash and organic non-sugars are determined by the usual methods of sugar analysis. The analysis of the melt of the technical period is computed from these analyses and the weights of material which they represent.

The refined sugar is weighed immediately after packing or, more generally, automatically weighed as packed. No analysis of the granulated sugar is made and its polarization is always taken as 100°. <sup>23</sup> Soft sugars are weighed, sampled and analyzed, as in the case of raw sugars, and a weighted average analysis is calculated for the period.

The barrel-sirup is sampled and measured by lots and the density and polarization of each is determined. The weighing may be conveniently done by means of the "Pneumercator." (See 377.) Weekly complete analyses are made of a composite sample and a weighted average analysis for the technical period is computed. At the end of every period—usually the technical periods include four weeks—an inventory of the material in process is made. All material containing sugar is measured and sampled. The density and purity of the samples are determined. The pounds of solids are computed from the volume of the material in cubic feet and the pounds solids per cubic foot, as indicated by the density. The solids multiplied by the purity give the sucrose. The solids less the sucrose give the solid impurities. In this way the total solids, total sucrose and total impurities in the stock are ascertained.

<sup>23</sup> This practice is equivalent to correcting the apparent polarization of the sugar for temperature error.

To compute these figures to available sirup and available granulated sugar, it may be assumed that all the impurities will go to make sirup of the same composition as that produced during the period.

Then:

$$\frac{\text{Lbs. solid impurities in stock} \times 100}{\text{Per cent solid impurities in sirup produced}} = \text{lbs. available sirup in stock; (1)}$$

$$\frac{\text{Lbs. available sirup in stock} \times \text{polarization of sirup produced}}{100} = \text{lbs. sucrose in sirup in stock. (2)}$$

Total sucrose in stock—sucrose in sirup in stock = lbs. available granulated in stock.

Example:

Assume a stock in process of 1,180,000 lbs. solids  
 850,000 lbs. sucrose  
 330,000 lbs. solid impurities.

Assume a barrel sirup produced as containing:

	Per Cent
Sucrose.....	34.2
Water.....	22.1
Solid impurities (ash, glucose, organic).....	43.7
	<hr/> 100.0

$$\frac{330,000 \times 100}{43.7} = 778,032 \text{ lbs. available sirup; . . . (1)}$$

$$\frac{778,032 \times 34.2}{100} = 258,261 \text{ lbs. sucrose in sirup in stock; . . (2)}$$

$$850,000 - 258,261 = 591,739 \text{ lbs. available granulated in stock.}$$

The increase or decrease in the available granulated in stock in process at the end of the period as compared to that at the beginning is added to or subtracted from the actual production of granulated for the period; the same calculation is made with regard to the sirup produced and the sirup in stock. These are *net* productions of sugar and sirup for the period.

These net weights produced are each divided by the weight of raws melted to give the net productions per cent melt. The analyses of the granulated, the soft sugars and sirup are calculated to per cents of the melts by multiplying each constituent of the analysis of each of these three products by the percentages of the product in terms of the melt. The various constituents of these analyses per cent of the melt can be totaled to give the analysis of the combined output of the refinery in terms of the melt. A comparison of this

analysis with that of the melt itself gives the increase or decrease of each constituent during the process of refining.

To illustrate this, let us assume a set of net yields for a refinery period and calculate the loss in sucrose for the period:

Polarization of the melt.....	96.20°
Net yield of granulated.....	90.58 per cent
Soft sugars produced.....	3.9 per cent at 88.8° polarization
Net sirup production.....	4.5 per cent at 34.1° polarization

Then,

Sucrose in melt..... 96.20

Sucrose in granulated per cent melt..... = 90.58

Sucrose in soft sugars per cent melt =  $\frac{3.9 \times 88.8}{100} = 3.46$

Sucrose in sirup per cent melt..... =  $\frac{4.5 \times 34.1}{100} = 1.53$

Sucrose in total product per cent melt..... 95.57

Sucrose lost in refining per cent melt..... 0.63

Following this same method of calculation, the gain or loss of invert sugar, ash, water and organic non-sugar may be traced.

**188. Chemical Control.**—The chemical methods employed in a refinery are similar to those used in factory work as described in the sections of this book devoted to analytical procedure. Clerget sucrose as well as direct polarization are determined on entering raws, barrel sirups and soft sugars in order to check the "sucrose balance" previously described.

The routine control of the refinery depends largely upon determinations of the purity of the materials in the successive steps of the process. The char-filter and pan-work are entirely controlled on a basis of purity. The number of these purity tests required day and night is so large that the work is usually conducted in a separate laboratory, on or near the vacuum-pan floor, by boys trained to do only this testing. This leaves the chemists free to do the analytical work involved in the technical control.

The dilute purity method of Casamajor is used in this work. The material—liquor, magma or sirup—is diluted to any convenient density, usually between 15° and 20° Brix, and the corrected Brix is determined. A part of the solution is clarified with Horne's dry subacetate of lead, if such preparation is necessary, and after filtration it is polarized directly. The polariscope reading multiplied by the factor corresponding to the degree Brix gives the purity coefficient. The factors are computed from the formula Factor =  $\frac{26.0 \times 100}{99.718 \times \text{sp. gr. } (20^\circ/20^\circ) \times \text{Brix}}$  which is Casamajor's formula as modified

by Rice for temperatures of 20° C. A table of these factors is given on page 493. The figuring is simplified by the use of the expanded Horne's table of purities for use in refinery control, given on page 494.

**pH Control.**—This has become an essential part of refinery control work. The various methods that are used are as given in Chap. XXII. The regulation of the reaction at the blowups has already been referred to (p. 161) and maintenance of pH within close limits is customary at this station. Tests of the pH of the materials going on and coming off the char are also frequently made and the pH of the water-extract of the revived char has been shown to be of the greatest value (p. 173). A careful study of the whole subject of pH control in a large refinery has been published by Blowski and Holven.<sup>24</sup>

**189. Special Analytical Methods.—Black Paste.**—A convenient routine method for determining the available phosphoric acid ( $P_2O_5$ ) in monocalcic phosphate-paste is as follows: Wash 10 grams of the paste into a 200-ml. flask with distilled water, breaking up the lumps with a rod. Make up to the mark, mix thoroughly by shaking and filter. Titrate 20 ml. of the filtrate (1 gram of the paste) with tenth-normal alkali, using methyl-orange as the indicator. This gives the acidity due to uncombined hydrochloric acid. The number of milliliters of N/10 alkali, with methyl-orange indicator  $\times 0.365$  = per cent free hydrochloric acid (HCl).

Titrate a second 20-ml. portion of the filtrate, using phenolphthalein indicator. This titration gives the total acidity due to the acid phosphates and the free acid. The calculations are made as follows: Number of milliliters N/10 alkali with phenolphthalein indicator—milliliters N/10 alkali, methyl-orange indicator  $\times 0.35$  = per cent available phosphoric acid ( $P_2O_5$ ).

**Boneblack: Preparation of the Sample.**—After thoroughly mixing the sample reduce it to 200–300 grams by subsampling. Pass a magnet through a thin layer of the sample to remove particles of iron that may have gotten into it from the retorts and filters. Grind about 100 grams of this prepared char in a porcelain mortar to a powder, all of which should pass through a 100-mesh sieve. The ground sample must be kept in a tightly stoppered bottle to prevent absorption of moisture.

**Boneblack: Moisture Determination.**—Heat 5 grams of the unground portion of the subsample during four hours at 110° C.

**Boneblack: Carbon and Insoluble Matter.**—Treat 2 grams of the ground char with 10 ml. of concentrated hydrochloric acid and 50 ml. of water. Boil gently for fifteen minutes, filter through a tared Gooch or alundum crucible and wash the residue with water to the disappearance of chlorides. Dry the crucible and contents at 100° C. and weigh; ignite to constant weight over the flame of a lamp.

Loss on ignition  $\div 2 \times 100$  = per cent carbon;

Residue after ignition  $\div 2 \times 100$  = per cent insoluble matter.

**Boneblack: Determination of Calcium Sulphate.**—To 25 grams of the powdered char, in a 250-ml. flask, add 25 ml. water and 100 ml. concentrated hydrochloric acid, gradually. Boil fifteen minutes. Add about 100 ml. water, and after cooling to room temperature, dilute to the mark with water, mix and

<sup>24</sup> Ind. Eng. Chem. Vol. 17 (1925), No. 12.

filter. Evaporate 200 ml. (= 20 grams of char) of the filtrate to about 150 ml. Proceed with the analysis as is described in page 396, using the concentrated solution obtained as above. The barium sulphate precipitate should be first washed by decantation. Calculation: Weight barium sulphate  $\div 20 \times .5833 \times 100$  = per cent calcium sulphate ( $\text{CaSO}_4$ ).

*Boneblack: Determination of Calcium Sulphide.*—To 25 grams of the powdered char in a 250-ml. flask add 0.5 gram of potassium chlorate, than 25 ml. of boiling water and follow this with 100 ml. concentrated hydrochloric acid, added very slowly. Proceed as in the determination of calcium sulphate, described above. Great care must be exercised in adding the acid, very slowly at first, in order that no sulphur be lost as hydrogen sulphide ( $\text{H}_2\text{S}$ ). The barium sulphate obtained in this analysis corresponds to the sum of the sulphide and sulphate in the char. Subtracting that already found for the calcium sulphate leaves the barium sulphate equivalent to the calcium sulphide. Calculation: (Total barium sulphate—barium sulphate derived from the calcium sulphate)  $\div 20 \times .3091 \times 100$  = per cent calcium sulphide ( $\text{CaS}$ ).

*Boneblack: Volumetric Determination of the Iron.*<sup>25</sup>—The following reagents are required:

(1) Standard potassium permanganate: Dissolve 4 to 5 grams of the salt in 1000 ml. of water. Check this solution against an iron solution of known strength, prepared as follows: Dissolve 2.5 grams of piano wire, or of the grade of iron wire that is prepared especially for standardizing, in a small quantity of hydrochloric acid, and dilute this solution to 250 ml. in a graduated flask. Use 50-ml. portions of this solution under the conditions of the analysis, as below, in standardizing the permanganate.

(2) Phosphoric acid and manganous solution: Dissolve 50 grams of manganous sulphate crystals in about 250 ml. of water, with the addition of a few drops of sulphuric acid; add 250 ml. of phosphoric acid solution of 1.3 specific gravity, followed in order by 150 ml. of water and 100 ml. of concentrated sulphuric acid. The phosphoric solution may be prepared from the 85 per cent acid ( $\text{H}_3\text{PO}_4$ ).

(3) Stannous chloride solution: Dissolve 30 grams of pure granulated tin in 125 ml. of concentrated hydrochloric acid, with heating. Solution is promoted by the addition of a few pieces of platinum foil. Dilute the solution with 250 ml. of water and filter it through asbestos. Add 250 ml. of concentrated hydrochloric acid and 500 ml. of water to the filtrate.

(4) Mercuric chloride solution: Dissolve 50 grams of the salt in 1000 ml. of water.

Proceed with the analysis as follows: Ignite 10 grams of the powdered boneblack and digest the residue with 30 ml. of concentrated hydrochloric acid, with gentle boiling during fifteen minutes. Filter the solution through a Gooch crucible and wash the residue thoroughly with small quantities of hot water. Heat the filtrate, contained in a large Erlenmeyer flask, to nearly boiling and add the stannous chloride solution to it drop by drop until the yellow color disappears. Add 60 ml. of the mercuric chloride solution, all at

<sup>25</sup> Adapted from Clowes and Coleman's "Quantitative Analysis," 5th ed., p. 206.

once, and mix well by shaking the flask; add 60 ml. of the phosphoric acid and manganous solution and 600 ml. of water.

The titration of the material prepared as above may be conducted in the flask placed over a white background or the solution may be transferred to a large porcelain dish. The flask, in the latter case, should be thoroughly washed and the washings added to the solution in the dish. Add the standard permanganate solution from a burette, with constant stirring, until the liquor assumes a faint pink color, which should disappear after three or four minutes' standing.

Make a similar titration of the solution prepared with the iron wire to ascertain the iron value of the permanganate solution. The percentage of iron in the char may be readily calculated from the data obtained in the two titrations.

*Boneblack: Calcium Carbonate and Phosphates.*—The calcium carbonate may be determined by the methods on page 394.

The percentage of phosphoric acid is of no particular significance, except when the spent char is to be sold on a basis of its fertilizing value. In this event, the customary methods of agricultural analysis are used.

*Boneblack: Thoroughness of the Revivification.*—The test of the efficiency of the revivification is of great value in the control of the kilns. Tests are made at very frequent intervals throughout the operating day on the char as it issues from the kilns.

To a measured volume of char add an equal volume of sodium hydroxide solution of 9° Brix. Heat the mixture to boiling for two minutes in a copper beaker and decant the soda through filter paper or cotton into a test-tube. A properly burned char will impart no color to the soda solution. A yellow or a brown color indicates a poor revivification (underburning), the depth of the color being directly in proportion to the amount of organic matter remaining in the black. An excess of sulphides will give a greenish cast to the solution.

René Baus<sup>26</sup> proposed the use of permanent standards made of acidified brom-thymol blue for the comparison of these tests. As modified since the publication, the standards are made as follows: To test tubes containing 35 ml. of 0.5 per cent acetic acid are added 0.2 ml., 0.4 ml., 0.6 ml., etc., up to 2.0 ml. of the 0.04 per cent brom-thymol blue used as pH indicator. These ten standards are numbered arbitrarily from 1 to 10, number 1 being the lightest and number 10 the brownest color. Soda tests are compared with these and the results may be reported numerically.

The pH of the water extract is possibly of greater importance than the soda test. (See work of Wayne, p. 173.) This is determined by shaking up the freshly burned char with neutral water, then filtering through paper or cotton that is free of acidity and determining the pH (generally on the phenolphthalein range). If the pH is above 8.4 (that is, pink to phenolphthalein) the alkalinity of the boneblack is satisfactory (though a pH as high as 9.0 is preferable). If below this the temperature of revivification should be raised, no matter whether the soda test shows colorless or not. Formerly, a pink test with phenolphthalein on the water extract of the char was considered as an

<sup>26</sup> Ind. & Eng. Chem. Vol. 19 (1926), No. 11.



indication of "over-burning," though many refiners recognized that moderate over-burning in this sense was a virtue rather than a fault. Now there is little possibility in ordinary kiln work of reaching the limits of true over-burning (pH of 9.6+).

**Boneblack: Grist Test.** Frequent determinations of the grain size of the boneblack are useful. A mechanical screen of the Ro-Tap type should be used, 100 grams being a convenient sample. "Hardness" tests on boneblack have been suggested by Knowles,<sup>27</sup> Horne and others based on the principle of determining the extent of grinding of the char in a ball mill under regulated conditions. The test is described by Knowles as follows:

For purposes of test, the sample of boneblack is sieved to obtain the 16 X 24 fraction. One hundred grams of this fraction are sieved on 24-mesh and 50-mesh sieves for ten minutes, using a Ro-Tap mechanical sieve shaker. The portions passing the 24-mesh and 50-mesh sieves are weighed. The three fractions are reunited and put in a ball mill (turnip-type) with 10 steel balls  $\frac{1}{4}$  inch diameter. After rotating the mill for fifteen minutes, or for 450 revolutions, should the speed be somewhat different than 30 r.p.m., the char is removed and resieved. The total net loss on 24-mesh has been termed the "shrinkage number," and the net gain through 50-mesh has been designated as the "discard number," it being assumed that in practice char finer than 50-mesh would be removed by screening.

Duplicate tests give results that seldom vary more than 0.5 for the shrinkage number and 0.2 for the discard number.

**Boneblack: Weight per Cubic Foot.**—Both the "loose" and "packed" weights are required. These weights are calculated from the apparent specific gravity of the char under the two conditions.

**Loose weight:** Fill a tared 500-ml. flask loosely with char. The flask should not be shaken or tapped during filling. The weight of this char divided by that of an equal volume of water gives the apparent specific gravity of the sample. Calculate the weight as below.

**Packed weight:** Proceed as before except that the flask should be shaken and tapped constantly while filling until no more char can be added to the flask below the mark. Multiply the apparent specific gravities by 62.5 to ascertain the weights of a cubic foot of the char.

**Boneblack: Decolorizing Power.**—By far the best test of a boneblack is its performance in actual use. Laboratory filtrations for comparing the decolorizing and adsorptive powers of chars may be carried out in cylindrical copper funnels of convenient dimensions, e.g., 4 inches in diameter by 15 inches high. Each funnel should be provided with a small cock at the bottom. The char should rest upon a perforated copper plate covered with cloth. The funnels or filters should be immersed in a water-bath, provided with suitable openings for the outlet-cocks, and should be filled to within a few inches of the top with the chars to be compared. The weight of the char should be the same in each of the filters.

A suitable solution for comparisons is prepared by dissolving a molasses-sugar to form a liquor of about 55° Brix clarified by filtration with kieselguhr as in the refinery. This liquor should be heated to about 165° F. and equal

<sup>27</sup> Ind. Eng. Chem. Vol. 19 (1927), No. 2.

portions of it should be added to each filter, little by little, or as under service conditions, to avoid forming air-pockets. After covering the char, the remainder of the liquor may be poured into the filter. Maintain the temperature of the water-bath at 160°–170° for several hours and then draw off the filtered liquor from the outlet-cocks. Compare the colors of the filtrates and unfiltered liquor and make complete analyses of each. The percentage absorption of color, ash and organic non-sugars, as well as the improvement in purity and the change in the glucose ratios may now be calculated for each char, using the constituents of the unfiltered liquor as a basis. A supply of a good grade of boneblack should be kept as a standard of comparison, if many such tests are to be made.

It is essential in all tests of this character that the conditions of the experiment shall be identical for all the samples of black to be compared. Certain points of the procedure must necessarily be arbitrary and the conditions can best be chosen to suit the particular requirements of the experiment. In so far as is possible, factory conditions as regards temperature, ratio of char to sugar, density of the liquor, etc., should be maintained. Freshly burned black should be used in these tests as the adsorptive power changes on cooling and exposure to the air.

**190. Examination of Refined Sugar.**—Chemical analysis of refined sugar is rarely needed since it polarizes so close to 100° (corrected for temperature) that the difference is immaterial for most purposes. The moisture is generally determined as a routine procedure, 0.02 per cent to 0.05 per cent being the limits of dry refined.

**Grist Test.**—Grain analysis is useful in controlling the pan work and keeping the commercial product within reasonable limits. Mechanical screening devices, such as the Ro-Tap, are convenient and give tests which check well with each other. A simple graphic method of reporting screen tests is shown in Fig. 53. Study of results of many tests showed that the significant figures were the amount of sugar staying on a 30-mesh screen (directly proportional to the coarseness of grain) and the amount of sugar going through a 50-mesh screen (inversely proportional to coarseness of grain). By representing two

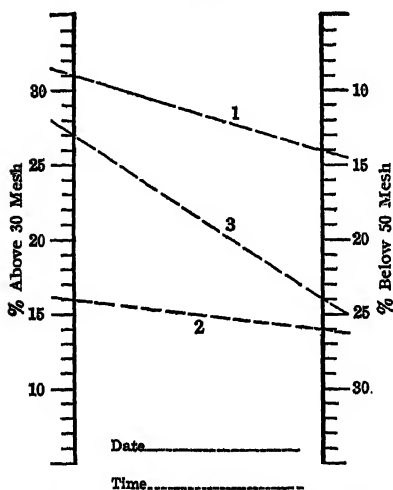


Fig. 53.—Chart for Recording Grist Tests.

vertical scales as shown, one direct and the other inverse, results may be reported as shown by the dotted lines. The sugar represented by line (1) had 31 per cent of coarse (above 30) and 16 per cent of fines (below 50) and the sugar is immediately seen to be a rather coarse grade of fine granulated. (True

*coarse granulated* is much larger.) Sugar (2) has 16 per cent on 30 mesh and 26 per cent through 50 mesh and is an "extra fine" granulated. Sugar (2) has 27 per cent on 30 and 24 per cent on 50 and is seen to be a badly mixed grain, i.e., much coarse and much fines mixed. Examination of the grain of refined sugar by the "projectoscope" described in Sec. 146 is frequently of value.

*Color of Refined Sugar.*—This is generally judged with the eye, by comparison under artificial "daylight light" with other sugar. If the sugars are placed in two small piles side by side and then flattened down by a piece of paper until they come together the line of demarcation will show otherwise imperceptible differences.

The so-called "blowup test" is also useful. It consists of examining a heavy liquor made from the sugar (2 parts sugar to 1 part distilled water, brought to a boil to expel air bubbles) for color and turbidity. A high-grade refined for candy making should show a water-white liquor when examined in a Nessler tube, while ordinary granulated sugar should be at most a faint golden yellow, with no tendency to grayish or greenish casts. A simple turbidiscopes by Horne and Rice which may be used for examining these and other sugar liquors for turbidity,<sup>28</sup> is described as follows:

The source of light is a good, nitrogen-filled, concentrated filament, incandescent lamp bulb, hung in a tin cylinder fitting quite closely around it and extending a little above the top of the lamp and about 1 cm. below the filament. About 1 cm. below this cylinder a second tin cylinder is attached by three soldered wires, and both are painted black on the outside and inside to prevent dispersion and reflection. Around each cylinder is a metal annulus perforated with holes to accommodate test tubes. At the bottom is soldered a broader annulus of tin to serve as a support for the test tubes. It can also serve as a base for the apparatus, but it is better to support it by the lamp socket, so that it can be easily turned and will be air-cooled. When the solution to be examined is put into a tube and set in the rack, there becomes apparent Tyndall's phenomenon of the reflection of light by the minute suspended particles, all viewed against a black background.

*Candy Test.*—This test has been used for a great many years to approximate the conditions of making hard candies. To 227 grams of the sugar add 87 ml. of distilled water in a copper casserole of about 750 ml. capacity. Heat over a naked flame so regulated that the total time of heating to 350° F. (177° C.) will be 21–23 minutes. The mixture is constantly stirred until the sugar is dissolved after which the stirring rod is removed. The solution should start to boil in 5–5½ minutes, after which the casserole is covered with a watch glass.

After heating fifteen minutes from the time the casserole was first placed over the flame, remove the watch glass, insert a thermometer and stir constantly with the thermometer until the temperature reaches 350° F. Remove the casserole from flame immediately and pour the contents rapidly on a cold slab. After the candy is brittle, it may be broken up and polarized. A comparison of the darkening and the drop in test of the "candy" caused by the heating are the points of interest to the candy-maker.

<sup>28</sup> Ind. and Eng. Chem. Vol. 16 (1924), No. 6.

This test came into some prominence in conjunction with a study of colloids in cane and beet sugar manufacture by Paine, Badollet and Keane of the Carbohydrate Laboratory of the Bureau of Chemistry.<sup>29</sup> They determined polarization, pH, surface tension, and reducing sugars on several sugars and their corresponding "candies" made by the test as well as the color of the candy. There were no very convincing findings as to the relationship between the various factors.

<sup>29</sup> The Planter. Vol. **73** (1924), Nos. 25-26.

# SUGAR ANALYSIS

## AND CHEMICAL CONTROL OF THE FACTORY

### CHAPTER XVI

#### SUGARS AND OTHER CONSTITUENTS OF THE CANE AND ITS PRODUCTS

**191. Sugars.**—In the manufacture of sugar from cane the sugars of importance in the analytical work are sucrose, dextrose, and levulose.

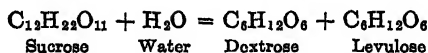
Sucrose, or cane-sugar, is the most important of these. Dextrose and levulose, and possibly other reducing-sugars, usually grouped with them by cane-sugar chemists under the name "glucose," are of importance on account of their influence in analytical and manufacturing processes. The sugars are classed chemically as carbohydrates. A table showing the more important chemical and physical properties of the carbohydrates is given on page 526. One of the physical characteristics of the greatest importance to the sugar chemist is the property of the carbohydrates of rotating the plane of a beam of plane polarized light.

**192. Sucrose, Saccharose, or Cane-sugar.**—This sugar is very widely distributed in the vegetable kingdom and in its pure state is the refined sugar of commerce. In a classification of the sugars it belongs to the disaccharides, derivatives of hexoses, and its formula is  $C_{12}H_{22}O_{11}$ . The commercial sources of cane-sugar are the sugar-cane, the sugar beet, the maple-tree, and certain palms. The sorghum-cane is often very rich in sucrose, rivaling the tropical cane, but the manufacture of sugar from this plant has not been a commercial success, though very large quantities of table-sirup are annually made from it, in nearly all parts of the United States.

Sucrose crystallizes in the monoclinic system, forming hemihedral anhydrous transparent crystals. The specific gravity of the crystals at  $\frac{17\frac{1}{2}^{\circ}}{17\frac{1}{4}^{\circ}}$  C. is 1.58046 (Gerlach). Sucrose is readily soluble in water and in diluted alcohol. It is practically insoluble in absolute alcohol, ether, chloroform and anhydrous glycerine. Dry sucrose, free of raffinose or other impurity, may be heated to a temperature of 120°–125° C. without discoloration it melts at a temperature of 160° C. Moist sucrose decomposes at temperatures above 100° C.

It has long been known that sucrose when warmed with acids changes its

chemical and optical character. The chemical change is expressed by the reaction.



This adding-on of water gives rise to the term "hydrolysis." The optical result is a change from right rotation to left rotation and on account of this reversal of the optical properties the process is termed "inversion" and the product, which is a mixture of equal parts of dextrose and levulose, is called "invert sugar." Certain ferments (the commonest being invertase, present in yeast and in the saliva) have the same property of hydrolysing or inverting sucrose. Sucrose itself is not directly fermentable but after the inversion the invert sugar is readily fermented. The inverting power of acids differs widely as is shown by the following table:<sup>1</sup> where the inverting power of hydrochloric acid is taken as 100.

Acid	Inverting Power, HCl=100	Acid	Inverting Power, HCl=100
Hydrobromic.....	111.4	Phosphoric.....	6.21
Hydrochloric.....	100.0	Citric.....	1.72
Nitric.....	100.0	Formic.....	1.53
Methylsulphonic.....	100.0	Malic.....	1.27
Sulphuric.....	53.6	Lactic.....	1.07
Oxalic.....	18.57	Acetic.....	.40

The speed of inversion is closely proportional to the electrical conductivity of the acid and its chemical affinity. Raising the temperature greatly increases the inverting power, the rate of increase being about the same for all acids.

Sucrose in common with many other substances has the property of rotating the plane of polarization of a ray of light. It rotates the plane to the right and is termed dextrorotatory. This property is utilized, as will be shown (see 198), in the construction of polariscopic apparatus, for the analysis of sugars.

**193. Dextrose.**—This sugar is also widely distributed in the vegetable kingdom, where it is found in mixtures with other sugars. Its chemical formula is  $\text{C}_6\text{H}_{12}\text{O}_6$  and it belongs to the monosaccharides (aldo-hexoses). Dextrose is always present in sugar-cane, and when sucrose is inverted, it and levulose are formed in equal quantities.

Anhydrous dextrose forms rhombic crystals whose melting point is  $144^\circ$ – $146^\circ$  C., and the hydrate forms crusts or transparent crystals which melt at  $80^\circ$ – $90^\circ$  C. Dextrose is manufactured on a large scale and sold as "corn" sugar. It has a sweetness of about two-thirds that of sucrose.

<sup>1</sup> Browne's "Handbook of Sugar Analysis," page 663.

Dextrose is readily soluble in water and in alcohol, the solubility in the latter varying with its dilution and temperature. It rotates the plane of polarization of light to the right, as the name would imply.

The chemical methods for the detection and estimation of dextrose, as well as other sugars, are based upon its property, in alkaline solution, of absorbing combined oxygen and reducing metallic oxides to lower oxides. The reaction utilized in analysis is the reduction of cupric copper in certain forms to insoluble cuprous oxide. For other properties of dextrose, refer to the table, page 526.

**194. Levulose.**—This sugar is usually associated with dextrose and sucrose in sugar-cane. It is widely distributed, and is often called fructose or fruit-sugar. Levulose is a monosaccharide, hexose (keto-hexose); it forms colorless, shining, crystals of the rhombic system, which are hygroscopic and until recently were considered difficult to produce. In 1926 Jackson, Silsbee and Profitt<sup>2</sup> published a method for producing it on a manufacturing scale from the Jerusalem Artichoke (*Helianthus tuberosus*). Former investigators had reported levulose as having a needle-like crystal but they found that when crystallized from a water solution, it always had the shape of a slightly tilted cube. Levulose is sweeter than sucrose, the relationship in sweetness being variously stated by different investigators from 103 per cent to 173 per cent that of sucrose. The crystals melt at 95°–105° C. The chemical formula of levulose is  $C_6H_{12}O_6$ . This sugar is very soluble in water and alcohol. It rotates the plane of polarization of light to the left, and is therefore termed laevorotatory. In mature cane the quantity of levulose is small as compared with the dextrose. Both these sugars are sometimes present only in faint traces, and occasionally the levulose is absent.

Even though levulose be absent or present in very small quantity in the juice it always appears in large proportion in defecation-process molasses. This increase of levulose is not entirely due to inversion and may occur when no sucrose has been inverted. Such molasses will often have a low direct polarization and a very high, true sucrose number.

The reappearance or increase of levulose is due to the action of alkalis and salts of alkalis with heat upon the dextrose. A part of the dextrose is converted into levulose. Likewise under similar conditions levulose may be isomerized and converted into dextrose. There are many references to this phenomenon in the chemical and technical journals.

**195. Invert Sugar.**—When sucrose is acted upon by acids and certain other reagents it combines with water and is converted into a mixture of equal parts of dextrose and levulose. The sucrose is said to have been hydrolyzed or inverted. This mixture of sugars is called invert-sugar.

The expressions "invert-sugar," "reducing-sugars" and "glucose" are used synonymously in the cane-sugar laboratories.

**196. Abnormal Constituents of Sugar-cane Products.**—There are a large number of compounds which, while not occurring normally in the juice of the

<sup>2</sup> Bur. Standards Sci. Paper 519.

<sup>3</sup> This article (196, 197) is included through the courtesy of Dr. Charles A. Browne, who prepared it at Dr. Spencer's request.

cane, yet occasionally make their appearance in the cane and its numerous products, as the result of fermentation or of destructive influences during the process of manufacture. These abnormal products may be roughly classified as follows: *first*, sugars and closely related derivatives of the same; *second*, the gums; *third*, acids; *fourth*, alcohols and esters; *fifth*, gaseous products. A few of these numerous compounds may be briefly mentioned:

*1st, Sugars and closely related derivatives of the same.*

Mannose,  $C_6H_{12}O_6$ , and the non-fermentable sugar glucose  $C_6H_{12}O_6$  have been reported by Pellet <sup>4</sup> in cane-molasses from Egypt. These two products are produced by the action of alkalis upon dextrose and levulose. They occur in cane-molasses in perceptible amounts only when an excess of lime has been used in the clarification.

Mannite ( $C_6H_{14}O_6$ ). This body is formed in considerable amount through the reduction of dextrose and levulose in certain fermentations of juices and sirups. Glycerol ( $C_3H_8O_3$ ) and Dimethylketol <sup>5</sup> ( $C_4H_8O_2$ ) have been reported in fermented molasses in small amounts.

*2d. The Gums.*

Dextran ( $C_6H_{10}O_5$ )<sub>n</sub>. This gum, a most common and troublesome enemy of the sugar-maker, is the product of various fermentations, such as that produced by the *Leuconostoc* (Froschlaich), and is of frequent occurrence in canes injured by freezing or by insect ravages. The formation of this viscous gum in canes soon renders them worthless for milling. Its high specific rotation (+200), three times that of sucrose, introduces a serious error into the polarization of cane-products, unless the gum is first removed by alcohol. Hydrolysis of dextran, with acids gives dextrose.

Levan. A gum found by Smith <sup>6</sup> and Steel in sugar-cane products in Australia and produced by the *Bac. levaniformans*. This organism was found to be very destructive in raw sugar; it causes a rapid inversion of sucrose and produces the slimy gum, levan, which has a specific rotation of -40. Hydrolysis of levan, with acids, yields levulose.

Cellulan. A gum found by Browne <sup>7</sup> in the tanks of a sugar-house in Louisiana. It is formed in certain fermentations of cane-juice and sirup, and consists of large leathery lumps insoluble in caustic alkali (distinction from dextran). The gum on treatment with boiling alkali yields a product giving all the reactions of cellulose (solubility in cuprammonium and blue coloration with zinc-chloride and iodine), and on hydrolysis with acids is converted into dextrose.

Mannan. A gum found occasionally in the sedimentary deposits of fermented juices and sirups. Hydrolysis of mannan, with acids, gives mannose.

Chitine ( $C_{12}H_{20}N_2O_{15}$ ?). This substance, which strictly speaking does not belong to the gums, was found by Browne <sup>7</sup> in large quantities in the scums of

<sup>4</sup> Report of the 5th International Congress of Applied Chemistry, 3, 383.

<sup>5</sup> Browne, *The Planter*, 1905, p. 237, vol. 34.

<sup>6</sup> *International Sugar Journal*, 4, 430.

<sup>7</sup> *The Planter*, 1905, p. 238, vol. 34.



hot-room molasses in Louisiana. It is of fungoid origin and on hydrolysis with hydrochloric acid yields an amine-sugar, glucose-amine ( $C_6H_{11}O_5NH_2$ ).

### 3d. *Acids.*

Formic, acetic, propionic, butyric, capric, and various other acids of the fatty series, have all been found among the fermentation-products of cane-juices and sirups. In addition to the above, lactic acid should be mentioned as of very common occurrence in juices, sirups, and molasses. The latter acid may be formed either by the action of lime upon the sugars of the juice during clarification, or through the agency of various organisms, as *Oidium lactis*, *Bac. lacticus*, etc.

Oxalic, glutaric, and other dibasic acids have also been reported as occurring in molasses in small amounts.

### 4th. *Alcohol and esters*

Ethyl, amyl, butyl, and other alcohols have been found in the fermented products of the sugar-cane. The combination of these alcohols with the various acids, acetic, butyric, etc., gives rise to numerous esters, the fruity odor of which is characteristic of many fermented cane-products. Ethyl sulphite and sulphide have also been found in fermented juices which have undergone sulfitation, and the objectionable odor (tufo) of these compounds is frequently noticeable in the spirits from tropical distilleries.

### 5th. *Gaseous products.*

Carbonic acid ( $CO_2$ ) is nearly always given off by cane products undergoing fermentation. In many fermentations a reduction takes place and in such cases hydrogen may be evolved. The explosion of hydrogen generated from the "sourwater," left standing in vacuum pans, has occurred at times, and on one occasion with disastrous effects.<sup>8</sup>

Another product in the reducing fermentation of juices which have been sulfured is hydrogen sulphide ( $H_2S$ ), and the odor of this gas from sulfured juice or sirup which has been left standing a long time is sometimes almost unbearable.

**197. Composition of Sugar-cane Molasses.**—The composition of sugar-cane molasses is very variable, being dependent not only upon the composition of the cane, but also, and to a much greater degree, upon the process of manufacture. The table on p. 196, condensed from a large number of analyses made at the Louisiana Sugar Experiment Station, gives the approximate composition of third molasses. (Browne.)

The xanthin bodies of cane-molasses are probably derived from the breaking up of the nucleo-proteids of the juice during clarification. A large part of the acid bodies of molasses, as melassinic, glucinic, and saccharinic, are not present in the juice, but are formed by the action of lime upon the reducing sugars in the clarification.

<sup>8</sup> La. Planter, 1890, 5, 243.

	Per Cent		Per Cent
Water.....	20.00		20.00
		{ Silica, $\text{SiO}_2$ .....	0.50
		{ Potash, $\text{K}_2\text{O}$ .....	3.50
		{ Lime, $\text{CaO}$ .....	1.50
		{ Magnesia, $\text{MgO}$ .....	0.10
Ash....	8.00	{ Phosphoric acid, $\text{P}_2\text{O}_5$ .....	0.20
		{ Sulphuric acid, $\text{SO}_3$ .....	1.60
		{ Chlorine, $\text{Cl}$ .....	0.40
		{ Soda, iron, etc., $\text{Na}_2\text{O}$ , $\text{Fe}_2\text{O}_3$ , etc.....	0.20
		{ Sucrose.....	32.00
Sugars.....	62.00	{ Dextrose.....	14.00
		{ Levulose.....	16.00
		{ Albuminoids.....	0.30
		{ Amids (as asparagin).....	0.30
		{ Amido acids (as aspartic)....	1.70
Nitrogenous bodies.....	3.00	{ Nitric acid.....	0.15
(Total N = 0.5 per cent)		{ Ammonia.....	0.02
		{ Xanthin bodies.....	0.30
		{ Other nitrogenous bodies....	0.23
Soluble gums.....	2.00	(Xylan, Araban, Pectin, etc.).	2.00
Free acids.....	2.00	{ Melassinic, Glutinic, Sacchar-	
Combined acids.....	3.00	{ inic acids, etc.....	5.00
Total.....	100.00		100.00

Molasses also contains a small quantity of carmelization product, the amount of these depending upon the temperature of evaporation and boiling.<sup>9</sup> Caramel is always formed by overheating cane-sugar and is a mixture of several dark-colored bodies of uncertain composition.

<sup>9</sup> Caramelan, Caramelen, Caramolin, *see* von Lippmann, *Chemie der Zuckerarten*, 3d ed., 1210.

## CHAPTER XVII

### OPTICAL METHODS IN SUGAR ANALYSIS

#### APPARATUS AND MANIPULATIONS

**198. The Polariscope.**—The instrument used in the quantitative estimation of sugars by the optical methods is usually called a polariscope. The names "polarimeter" and "saccharimeter" are also sometimes used.

Sucrose, in common with many sugars and other substances, has the property of rotating the plane of polarization of a ray of light. Advantage is taken of this property in the construction of the polariscope.

The polarization of the ray of light is accomplished by passing it into a Nicol prism. This prism is made from a rhombohedron cut from a transparent crystal of Iceland spar. The end surfaces of the prism, Fig. 54, are ground off so that the acute angles are each  $68^\circ$ . The prism is then cut into two parts, *A* and *B*, through the obtuse angles, and the surfaces are polished and cemented together again, in their original positions, with Canada balsam. Other forms of the prism are used, but this description will answer for the present purposes.

On passing a ray of light into this prism it is separated into an ordinary ray, which is reflected from the prism by the balsam cement, and an extraordinary or polarized ray, which passes through the polariscope and upon whose properties the construction of the instrument depends.

Confining our remarks to three sugars of most interest to the cane-sugar chemist, it should be noted that sucrose rotates the plane of polarization to the right and is therefore termed a right-hand sugar. The expressions dextrorotatory and dextrogyratory are also applied to sucrose. Dextrose also rotates the plane to the right. Levulose rotates the plane to the left and is laevorotatory, or a left-hand sugar. Quartz is of two kinds, right and left hand, and is used in certain types of polariscopes to compensate for the rotation due to sugar.

The power of a sugar to rotate the plane of polarization differs for different sugars. The number expressing this is termed the specific rotation or specific rotatory power, and is the rotation in angular degrees which a calculated 100 per cent solution of the pure substance 100 mm. long gives to the plane of polarized light. The expression  $[\alpha]$  is used to indicate specific rotation and as it depends upon the wave-length of light employed and the temperature at

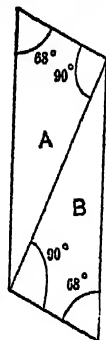


FIG. 54.  
Nicol Prism.

which it is taken these must be specified. The bright yellow line of sodium-light, known as the *D* line, is the light almost universally used and the standard temperature is 20° C.; the symbol for specific rotatory power under these conditions being  $[\alpha]_D^{20}$ .

It will be seen that when a ray of polarized light is passed through a solution of any given sugar the amount of rotation varies with the strength of the solution, the length of the column, the character of the light and the temperature. By having a fixed length of column, standard temperature and class of light the rotation then becomes a function of the concentration of the sugar in the solution.

These optical properties of sugars, Iceland spar, and quartz are utilized in the construction of the polariscope, and keeping them in view the descriptions of the various types of instruments, as given farther on, will be readily understood.

Polariscopes were formerly of two classes, viz., shadow and transition tint instruments but the tint polariscopes have long been obsolete. The shadow instruments may be divided into polariscopes using white light, as from an electric lamp, and those requiring a monochromatic light, the yellow ray. The former are usually employed in commercial work and the latter in scientific investigations.

A brief description of the polariscopes in general use will be given in the following paragraphs and will be sufficient for the purposes of this book. The reader is referred to handbooks of the polariscope for more detailed descriptions of the theory and construction of the instruments.

**199. Half-shadow Compensating Polariscope (Schmidt & Haensch).—**The optical parts of this instrument are indicated in Fig. 55. The polariscope shown in Fig. 55 is of the single compensation type.

At *P* there is a slightly modified Jellet-Cornu Nicol prism, at *L* is a plate of laevorotatory quartz, at *E* is a quartz-wedge movable by means of the screw *A*, and at *D* is a quartz-wedge, fixed in position, to which is attached the vernier. The scale upon which to note the distance the quartz-wedge *E* has been moved, in compensating for the rotation of the plane of polarization due to interposing an optically active body, is attached to the wedge. The scale is graduated, for technical work, to read percentages of cane-sugar. These quartz-wedges are of dextrorotatory quartz.

The parts *D*, *E*, and *L*, constitute the compensating apparatus, i.e., that compensates for the deviation of the plane of polarization, as explained above. The substance to be examined, dissolved in a suitable solvent, is placed in the observation-tube, as shown in the figure. At *H* is the analyzer, a Nicol prism; at *F* is the telescope used in observing the field, and at *M* are the telescope and reflector for reading the scale. The two lenses at the extreme right are for transmitting the rays of light from the lamp in parallel lines to the Nicol prism, forming the polarizer.

The instrument described above is of the single-compensation type. A double-compensating instrument is shown in Fig. 56. The polariscope differs from that already described in having two sets of quartz wedges of opposite optical properties and two scales and verniers. The arrangement of double-

compensating wedges is shown in the optical parts of the Bates-Frič instrument, Fig. 59.

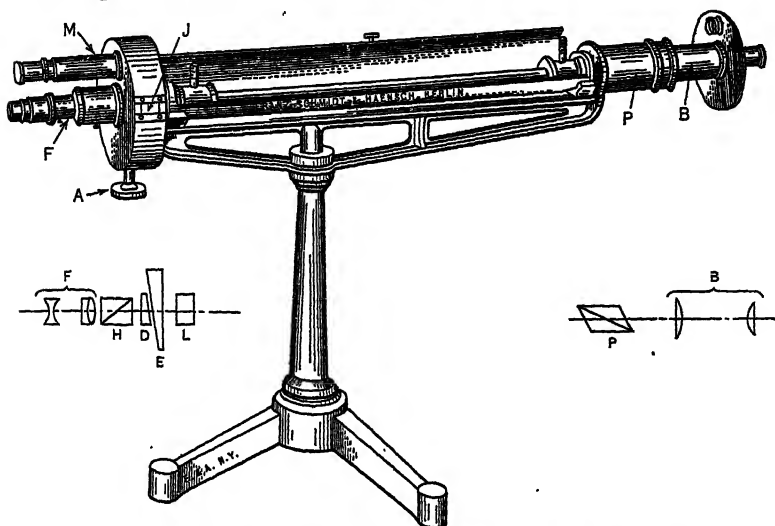
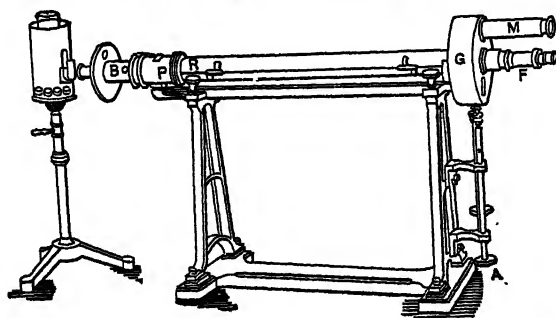


FIG. 55.—Schmidt and Haensch Single Compensating Polariscopes.

The field of vision of the above instruments, when set at the neutral point, is a uniformly shaded disk. If the milled screw, controlling the compensating wedge, be slightly turned to the right or left, one-half of the disk will be shaded



*Schmidt and Haensch.*

FIG. 56.—Double Compensating Polariscopes.

and the other light. It is from this half-shaded disk and the compensating wedges that this instrument takes its name.

The optical parts of all modern instruments are protected from exposure by the cap G, Fig. 56, and by plain glass plates. This protection of the optical parts is especially important in the tropics, where, owing to some

peculiar climatic condition, the lenses often become coated with an opalescent film that can only be removed by polishing. These plates also prevent minute spiders and other insects in the tropics from damaging the instrument. The scale is lighted from the lamp, the light being reflected by means of a prism and mirrors. The telescope *F* is focused by a screw. In the more modern instruments the screws for adjusting the position of the quartz wedges have long stems so that the hand of the observer may rest upon the table. A glass cell filled with a 3 per cent solution of bichromate of potassium is placed at *B* and serves as a ray filter.<sup>1</sup> This ray filter should always be used, except for dark-colored solutions. The heavy trestle support of Fig. 56 is preferable to the tripod form of Fig. 55 and is used in most modern polariscopes.

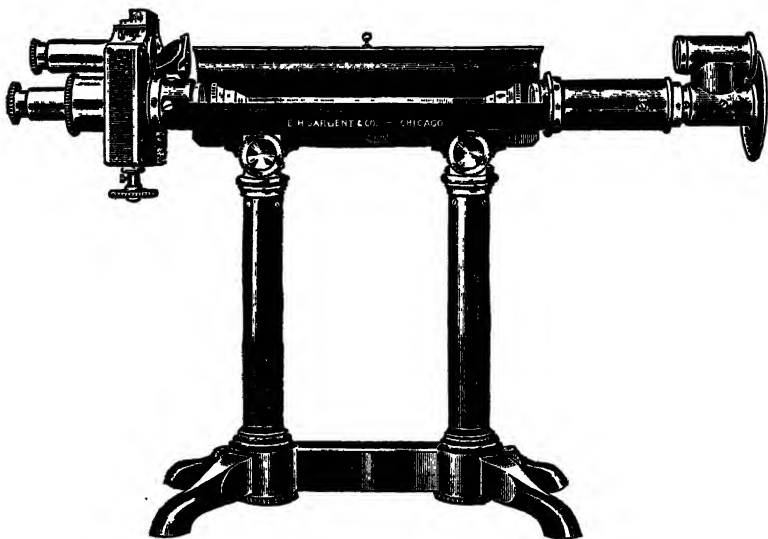


FIG. 57.—Fric Single Compensating Polariscope.

**200. Half-shadow Polariscope with Glass Scales (Josef-Jan Fric).**—The instrument shown in Fig. 57 has single-wedge compensation for use with white light. The optical parts are enclosed in a metal case for protection from dust. The scales and verniers are engraved on glass and are lighted by a part of the polarized rays which are reflected upon them.

Messrs. Fric also make a quartz-wedge polariscope with adjustable sensibility (Fig. 58), designed for the U. S. Bureau of Standards by Frederick J.

<sup>1</sup> This strength of solution assumes a cell 3 cm. in length. For other cell lengths, the product of the length by the percentage strength of the solution should be 9. According to Schönrock the use of a bichromate ray filter makes a difference of  $0.12^\circ$  in the polarisation with a white light instrument. With the filter he obtained a reading of  $100^\circ$  and without it  $100.12^\circ$ . (Circular No. 44, Bureau of Standards; *Proceedings of the Bureau of Standards*, 1904, pp. 521-558.)

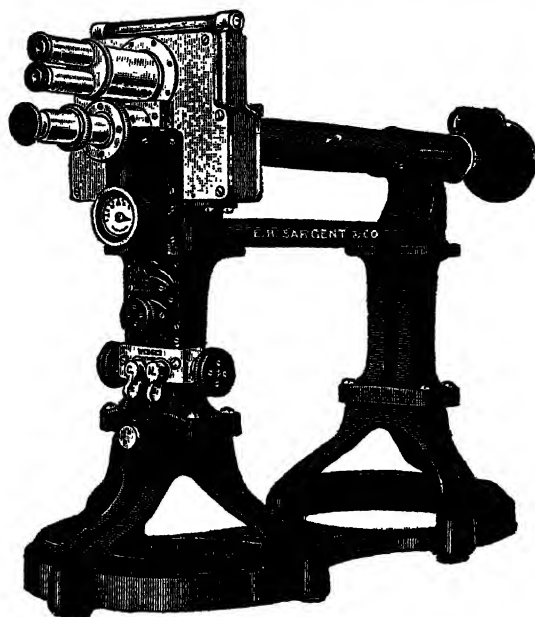


FIG. 58.—Bates-Frič Polariscopes.

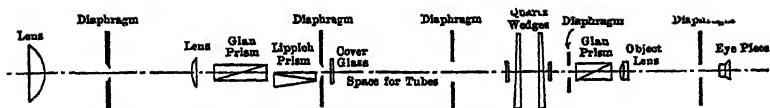
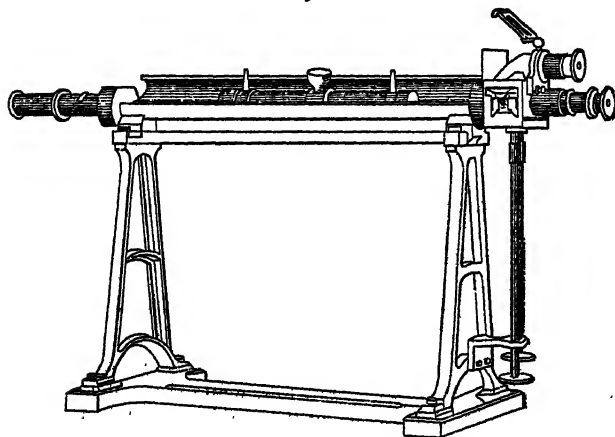


FIG. 59.—Optical System of Bates-Frič Instrument.



Bates. This instrument is double quartz wedge compensating and has a Lippich polarizing system. Readings may be accurately made to  $0.01^\circ$  sugar. The Frič instruments were among the first to be graduated according to the specification of the International Commission at  $20^\circ \text{C}$ . for use with the normal weight of 26 grams with the 100 true cubic centimeter flask at  $20^\circ \text{C}$ . All makers now conform to these specifications. The optical system of the Bates-Frič is shown in Fig. 59 and illustrates the arrangement of the quartz wedges in a double-wedge compensating polariscope.

**201. Half-shadow Polariscope (Julius Peters).**—This instrument, Fig. 60, is double-compensating. It is similar in principle to the other compensation polariscopes. The optical parts are protected by glass plates from the action of the atmosphere. A dust-cap, not shown in the figure, protects the optical parts at the front end of the instrument.

The quartz wedges are not mounted in brass as is usual, and it is claimed are not exposed to pressure or strain with the expansion of metal. As is well known, the rotation of quartz is changed by pressure.

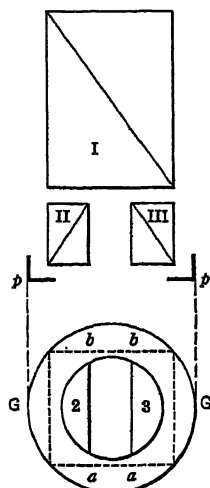


FIG. 61.—Prisms in Triple-Field Polariscope.

**202. Triple-field Polariscope (Schmidt & Haensch).**—This instrument differs from those already described in having two small Nicol prisms placed in front of the polarizer, as shown in Fig. 61. — The field of the instrument is divided into three parts, 1, 2, and 3 of the diagram. This figure shows the arrangement of the Nicol prisms (I, II, II), and a diagram of the field. When the instrument is set at the neutral point, the field is uniformly shaded; in other positions 1 is shaded and 2 and 3 are light or *vice versa*.

This arrangement permits a very high degree of accuracy in the adjustment of the field, in polariscopic observations, provided that the instrument is in perfect adjustment. It has been found, however, that minor differences in the two outer sections of the field are apt to develop and that the observer compares the central division with one of these sections only, making in effect only a double field. The triple-field type for this reason has not found general favor.

**203. Bausch and Lomb Polariscope.**—This instrument was designed after obtaining the opinions of many prominent sugar chemists as to the requirements for a high-class polariscope for industrial use, and it embodies several new features for convenience and cleanliness as well as for accuracy. It is single-wedge compensating with a scale range from  $-30^\circ$  to  $110^\circ$  and is equipped with either the Lippich double prism polarizer or the Jellet single prism type. The first system is very sensitive and the dividing line is practically invisible, but it is fragile and subject to disintegration, while the Jellet polarizer is less sensitive but not so easily put out of adjustment. The latter system (single



prism) is advocated for industrial laboratories. The diagram of the optical parts with Jellet polarizer is shown in Fig. 62, this figure illustrating in considerable detail the construction of a single-wedge polariscope. The scale is of glass, with the vernier and scale lines overlapping, which facilitates exact reading and the estimation of fractions of tenths. The scale field and the polarized field are illuminated from the same light source and the light to the scale passes through a filter similar to that used in the observation field, making both fields of the same color and intensity. In place of the bichromate cell is a ray filter of glass having the same optical properties, and which may be thrown out of the field by a push-rod when dark-colored solutions are read.

**204. The Polariscopic Scale. Ventzke Scale. The Normal Weight.**—Polariscope scales are divided to read either circular degrees or percentages of cane-sugar or both. Commercial instruments usually have only the Ventzke or cane-sugar scale.

The Ventzke or cane-sugar scale is so divided that, if a certain weight of the material be dissolved in water and the solution diluted to 100 cc. or ml. and observed under standard conditions in a 200-mm. tube, the reading will be in percentages of cane-sugar, or if pure sucrose be used, the reading will be 100. (See reference to Bates-Jackson Scale below.) The weight required under these conditions to give percentage readings is termed the "normal weight" or the "factor" of the instrument. In commercial work, especially in the polarization of sugars, the divisions of the cane-sugar scale are usually termed "degrees."

The normal weight for all modern instruments, as adopted by the International Congress of Applied Chemistry, is 26 grams. The flask used with this normal weight must hold 100 true cubic centimeters (milliliters)<sup>2</sup> of solution at 20° C. The solution must be prepared and the observation be made at 20° C. to secure the greatest accuracy.

The relationship between the older normal weight of 26.048 dissolved in 100 Mohr's cubic centimeters at 17½° C. and the present one of 26 grams in 100 ml. (true cubic centimeters) at 20° C. is extremely close. Exact mathematical correspondence would have been secured if 26.0082 grams<sup>3</sup> had been chosen instead of 26 grams but this difference of less than 1 mg. is quite evidently negligible in polariscopic work.

**Bates-Jackson Scale.**—In 1916 Bates and Jackson<sup>4</sup> published a report of experiments with pure sucrose repeatedly recrystallized from water *in vacuo* showing that the polarization of 26 grams of sucrose in 100 ml. of water at 20° C. was 99.895 on the Ventzke scale. This new value has been adopted by the Bureau of Standards in the standardization of quartz plates and saccharimeters, and because of this, Government polarizations (*e.g.*, for duty purposes) are always about 0.1° higher than those made on the same sugars in commercial or industrial laboratories.

<sup>2</sup> The Bureau of Standards has requested that the true cubic centimeter be called a milliliter (ml.) and this is followed in this book wherever accurate measurements are specified.

<sup>3</sup> Browne. Handbook of Sugar Analysis (N. Y., 1921), p. 114.

<sup>4</sup> B, S. Scientific Paper, No. 268.

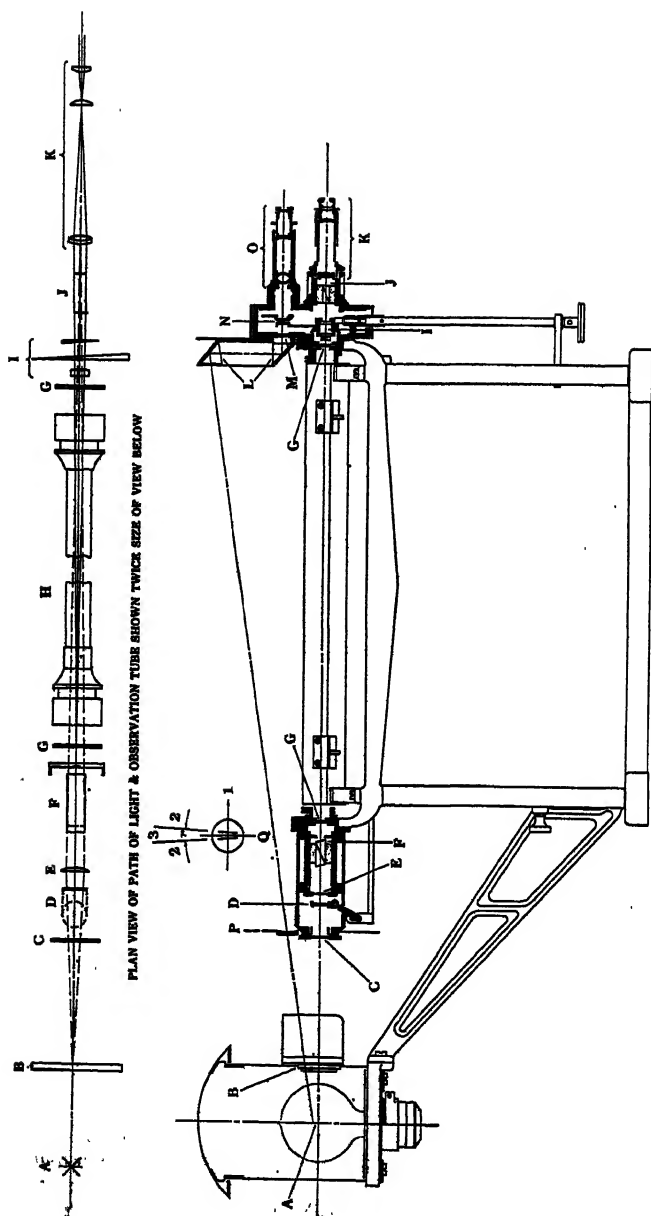


Fig. 62.—Diagram of Bausch and Lomb Polariscopes.

A, Light Source; B, Ground Glass Lamp Window; C, Cover Glass; D, Glass Filter; E, Replacing Bichromate Cell; F, Polariser; G, Splash Glass; H, Observation Tube; I, Quartz Compensation Tube; J, Analyser; K, Scale Illuminating System; L, Condensing Lens; M, Scale Reading Eyepiece; N, Scale and Vernier Plates; O, Glass Filter in Scale Illuminating System; P, Photometric Field as Seen from Eyepoint; Q, Plane of Polarization of Analyzer; R, Plane of Polarization of Semi-fields; S, Half Shade Angle.

Browne and Zerban<sup>5</sup> corroborated these figures of Bates and Jackson in work done at the Bureau of Chemistry and the New York Sugar Trade Laboratory respectively, finding that "100° point" on the Ventzke scale is actually 99.90. They conclude that "in working with refined sugars, high-grade sirups, and other products of high purity, where there are no counterbalancing plus errors due to the volume of lead precipitate and to other causes, it would be more accurate to employ a normal weight of 26.026 grams than the present weight of 26 grams. With products of low purity, however, where the counterbalancing plus errors may equal or exceed the minus error due to the graduation of the scale, the introduction of a corrected normal weight or of a correction factor will increase instead of diminish the errors of observation."

Until some action is taken by some of the associations governing official methods the present normal weight of 26 grams and the Ventzke scale will continue to be used in commercial work.

**205. Manipulation of a Polariscopes.**—Methods of preparing the solutions will be described in 209. Having dissolved and clarified the normal weight of the material to be polarized, fill an observation-tube with a portion of the solution and place it in the trough of the instrument, and pass the light from a suitable lamp through it.

The observer with his eye at the small lower telescope of the instrument will notice that one-half of the disk is shaded more than the other, provided the instrument is not set at the neutral point. The vertical line separating the half-disks should be sharply defined, and if not, the ocular should be moved backwards or forwards until a sharp focus is obtained.

The observer should now turn the milled screw, which moves the quartz-wedge compensator, until the field appears uniformly shaded, then read the scale as directed in page 206.

In making the observation the eye should be in the optical axis of the instrument, and should not be moved from side to side.

A little practice will enable the operator to detect very slight differences in the depth of the shadow or tint and to attain great accuracy in this manipulation.

The manipulation of the triple-field polariscopes is as described above, except that the field is in three sections.

The double compensating polariscopes are provided with two scales, in the older instruments, one graduated in black and the other frequently in red. The black scale is operated by the black screw and the red by the brass one. For ordinary work the red scale is set at zero and the field is equalized with the black screw.

Since the abandonment of ivory scales on account of their length changing with certain atmospheric conditions, metal or glass scales are usually used, and are marked to indicate whether for right or left readings.

To make an invert reading, *i.e.*, one with a laevorotatory or left-hand sugar, the black, or right-hand scale should be set at zero and the other scale used. The readings should be recorded with the algebraic sign minus. With the

<sup>5</sup> Jour. Ass'n. Off. Agr. Chemists, Vol. XI (1928), No. 1.

single-compensating instruments the direct and invert readings are made on the same scale, the graduations extending both sides of the zero.

Having equalized the field of the polariscope as already described, the scale is to be read. The method of reading the scale is best shown by an example. Let the position of the scale and vernier be as shown in Fig. 63. The zero of the vernier is between 30 and 31 of the scale; record the lower number and note the point at which a line on the vernier corresponds with one on the scale, that is, if extended would coincide with the line on the scale, in this case at 7 of the vernier. Enter this number in the tenths place and the completed reading is 30.7. If the zero of the vernier falls directly opposite a line of the scale, the reading is a whole number. The divisions to the left of the zero of the vernier are only needed in single compensating instruments and are for making invert readings.

If the normal weight of sucrose, or of a substance containing sucrose and no other optically active substance, has been dissolved in water and diluted to a volume of 100 ml., and a 200-mm. observation-tube has been used, and the observation is made at the temperature at which the instrument was standard-

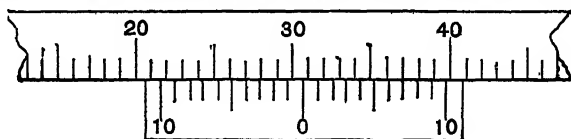


FIG. 63.—Diagram of Vernier Scale.

ized (now generally 20° C.) the reading on the sugar scale is the percentage by weight of sucrose in the material. The readings must be corrected for other weights than the normal, for other volumes than 100 ml., and for other tube lengths than 200 mm.

**206. Polariscope Lamps.**—Compensating half-shadow instruments require a strong white light.

The most efficient polariscope illumination in a factory having twenty-four-hour electric service is the nitrogen-filled concentrated-filament Mazda lamp of 100 watts capacity. This light is so very intense that a ground-glass plate should be interposed in polarizing light-colored solutions.

It has been found that there is considerable momentary variation in the voltage of factory current which may cause appreciable change in the intensity of the light and, consequently, errors in the polariscope readings. This may be obviated by having a rheostat and voltmeter located in the lamp circuit and reducing the voltage to a fixed point with the rheostat (*e.g.*, 100 volts for a 110-volt current). The voltmeter and rheostat should be located in full view of the operator (the upper, forward part of the polariscope compartment is convenient) in order that the voltage may be accurately adjusted for each reading.

Storage batteries and automobile headlight lamps are practicable where direct current is available for recharging. The Edison storage battery is simple to charge and requires little attention. The storage battery gives a fixed

voltage and a good light, but the ordinary current with adjusted voltage as described above is preferable.

Acetylene lamps provide a strong and convenient source of light, probably next to the concentrated-filament lamp in efficiency. The Welsbach lamp, burning gas, alcohol or gasoline, give a very good light.

Messrs. Schmidt & Haensch equip many of their instruments with small electric lamps operated from small secondary batteries. The lamp is provided with a special socket attached directly to the instrument. This arrangement of the lamp is not entirely satisfactory in practical experience. Kerosene lamps may be used and should have duplex burners. Electric and acetylene lamps and alcohol or gasoline Welsbach lamps are now so easily obtainable on plantations that they should be given preference in the order named over the kerosene light.

It is quite essential that the position of the light with respect to the polariscope be fixed. The intensity of the light should be as constant as possible, and if changed, the zero reading observation should be verified.

**207. Adjustment of the Polariscope.**—The scale of the instrument is the only part liable to get out of adjustment. To test this adjustment, place a polariscope tube filled with water in the trough of the instrument and make an observation. This observation may be made without the tube, but the adjustment on the zero is not so readily made as with the water. If the scale is properly adjusted the reading should be zero.

The method of adjusting is the same for all modern compensating polariscopes. A micrometer-screw, generally placed at the right-hand side of the dust-cap enclosing the forward optical parts of the instrument and turned by means of a key, is arranged to move the vernier a short distance. The field is equalized as usual, but with water in the observation-tube, and the vernier is moved by the micrometer-screw until the zero lines of the scale and vernier coincide with one another. The scale is now moved through several degrees by the milled screw and the field is again equalized as before, and if the zero lines do not coincide the vernier is to be again adjusted. These manipulations are repeated until the zeros coincide in several successive observations. These adjustments are very fatiguing to the eye, which should be rested a short time before making the final observations.

Certain compensating polariscopes, especially of the older models, are exceedingly sensitive to changes in the position or intensity of the source of light. It is advisable to follow the maker's directions as to the position of the lamp with regard to the instrument and arrange the latter so that it cannot be jarred out of place. The distance of the lamp from the instrument is usually 15 to 20 cm. The position the lamp should occupy should also be marked, that it may be properly replaced if disturbed, and the intensity of the light should not be changed after adjusting the instrument until the observation has been made. A change in the position or intensity of the light, with certain instruments, will sometimes cause an error of  $0.5^\circ$  or more. (See also p. 206.)

It is advisable to have quartz control-plates for checking the adjustment of the instrument and the correctness of the scale. Standardized plates of the highest accuracy may be obtained from the makers of polariscopes, and in use

take the place of the observation-tube and a standard solution of pure sucrose. One plate should read approximately  $96^\circ$  and a second about  $60^\circ$ , as these parts of the scale are used in the most important polarizations.

It is well to have the quartz plates standardized by the Bureau of Standards but it should be noted that the Bureau uses the Bates-Jackson scale (204) which is about  $0.10^\circ$  higher than the Ventzke scale; i.e., a plate marked  $98.50^\circ$  on the Ventzke scale will be certified as close to  $98.60^\circ$  by the Bureau of Standards.

The scale may be tested with a control-tube made by Schmidt & Haensch and shown in Fig. 64. The funnel *T* of the control-tube is filled with a sugar

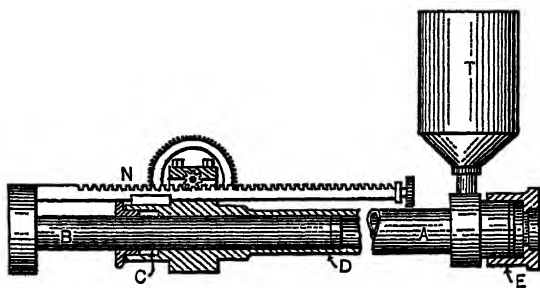


FIG. 64.—Control Tube.

solution, which flows into the tube as it is lengthened by turning the milled screw. The tube length is read on the scale *N*. A full description of the use and value of this instrument is given by Browne (Journal Industrial Engineering Chemistry, Vol. 12 (1920), No. 8).

The committee of the Fourth International Congress of Applied Chemistry also recommends the use of pure cane sugar for testing the polariscope. The preparation of pure sucrose is too exacting a process for the average laboratory. (See Bates and Jackson, Bur. Standards Paper No. 268.)

The analyzer and polarizer should not be removed from the instrument or adjusted except by an experienced workman, and in the event of an accident to the polariscope it should be returned to the dealer for repairs.

All parts of the polariscope should be kept very clean, especially the exposed parts of the lenses; these should be occasionally cleansed with alcohol and wiped with old linen.

**208. Observation Tubes.**—The usual tubes of glass are shown in Fig. 65. The upper tube has screw-caps and the lower the Landolt slip-cap. The slip-cap is designed to prevent undue pressure upon the cover-glass. The tubes of the French Laurent instrument are supplied with slip-caps that have bayonet-catches. A coiled spring is arranged inside the cap to bear upon the cover-glass and hold it in position, without unnecessary pressure.

Metal observation tubes of the Bates type, Fig. 67, are now used in many laboratories for all work except acid-inversion solutions, for which water-jacketed glass tubes, Figs. 71, 72, 73, are generally needed.

Tubes of the type shown in the illustration, Fig. 65, must be completely filled with the solution to be polarized. The cover-glass should be slipped onto the tube, pushing off the surplus liquid. The glass body of the filled tube should not be touched with the hands, since the warmth will cause

striations to form in the liquid. These prevent an immediate observation. In the event of striations, the tube should be left in the trough of the polariscope until the field clears.

*Observation Tube with Enlarged End.*—This tube is shown in Fig. 66. In using this tube, it is nearly filled with the solution to be polarized, leaving room for a small bubble of air, which rises to upper part of the enlargement. This arrangement obviates the necessity of excluding air-bubbles, and facilitates filling the tube.

*Bates' Observation Tube.*<sup>6</sup>—"In order to overcome the prevalent defects in the theoretical design, as well as secure a tube suitable for severe usage, this Bureau (U. S. Bureau of Standards) has brought out the Bates type of

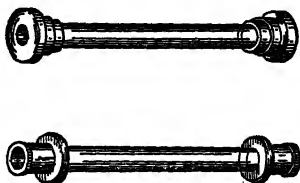


FIG. 65.—Observation Tubes.



FIG. 66.—Polariscope Tube with Enlarged End.

tube shown in Fig. 67. It will be observed that the weight is carried upon two shoulders, which are integral parts of the tube and not upon the caps, thereby eliminating all danger from turning while in the trough of the instrument. The bore is 9 mm., permitting the utilization of the full aperture of the polar-



FIG. 67.—Bates Metal Tube.

izing system. This also reduces to a minimum the light depolarized by reflection from the walls of the tube. The field of the instrument thus appears for the first time as a bright, sharply defined circle with no overlying haziness, and the reading can be made with increased accuracy. Both ends are enlarged

with all the attendant advantages, yet but one size of cover-glass and washer is required. The walls are unusually heavy, eliminating all danger from bending."



FIG. 68.—Polariscope Tube with Side Tubule.

*Observation Tube with Side Tubule.*—The tube shown in Fig. 68 is very convenient in general sugar-analysis. The cover glasses need rarely be removed. This arrangement reduces the risk of error by compression of the cover glasses. The tubes should be frequently cleaned with diluted acetic acid.

<sup>6</sup> Copied from Circular No. 44, "Polarimetry," U. S. Bureau of Standards, p. 39.

*Pellet's Continuous Tube.*—This tube, Fig. 69, is designed for very rapid polarization and is especially adapted to the use of laboratories for beet-seed selection and the purchase of raw material on a basis of its sucrose content. The Pellet tube is also convenient in the control of the char-filters in sugar refineries.

The Pellet tube provides for the rapid change of solutions without removing it from the trough of the polariscope. The tube is so arranged that it may



FIG. 69.—Pellet's Continuous Tube.

form a part of a syphon by connecting rubber tubing to the tubules. The short leg of the syphon terminates in a glass tube which is dipped into the new solution and a

pinch-cock on the long leg is then opened. The incoming liquid displaces the previous solution. This arrangement should be used only with solutions of approximately the same polarization and not when scientific accuracy is desired. The funnel arrangement illustrated in Fig. 70 should be used when accuracy is necessary, and in its use liberal quantities of the solution should be used to wash the funnel.

The Pellet tube should be washed with distilled water before a period of idleness and should be left filled with water.

A modification of this tube by Spencer is shown in Fig. 70 and differs from Pellet's design only in having four grooves to distribute the solution instead of one. The funnel directs the solution into an annular canal, which connects by separate openings with each of the four grooves shown at the end of the tube. The solution is delivered against the inner surface of the cover-glass,



FIG. 70.—Pellet's Tube (Spencer Modification).

and by a similar set of grooves and canal at the opposite end of the tube the displaced solution flows through a bent tube to the waste-jar. The observation cannot be made until the previous solution is entirely displaced. So long as any of the old solution remains the field will not be clear.

The Pellet tube is possibly accurate enough for raw sugar polarizations, if used with caution, but it is not recommended. The best practice for such accurate work is to have as many individual tubes as there are sugars to be polarized in each batch of tests, then fill each tube placing it in a rack. The rack of filled tubes is then placed in the polariscope compartment and allowed to stand so that temperature equalization is assured after which they are polarized and recorded.

*Landolt's Inversion Tube: Wiley's Modification.*—This tube, Fig. 71, is arranged for the control of the temperature of the solution under observation,



especially in the Clerget double-polarization method. The glass observation tube is enclosed in a metal jacket through which water is circulated while polarizing. A side tubule, enlarged to funnel shape, is provided for use in filling the tube and in taking the temperature of the solution. A centigrade thermometer graduated to fifths of a degree should be used.

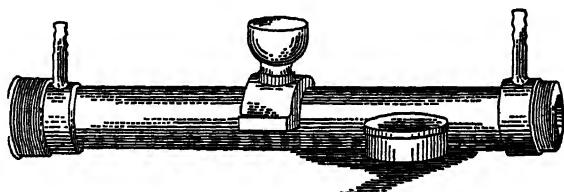


FIG. 71.—Landolt's Inversion Tube.

Wiley devised the desiccator-caps shown in Fig. 72 to prevent the condensation of moisture upon the cover-glasses at temperatures below the dew-point of the air. These caps are of hard rubber and have a central tube surrounded by fused calcium chloride to dry the air within the cap. A recent form of inversion tube (Fig. 73) has baffle plates to insure proper circulation of



FIG. 72.—Landolt Tube with Wiley's Desiccator Caps.

cooling water; the thermometer is offset to avoid interference with the light to the polariscope scale; the stopper is of ground glass with the thermometer cemented into it, the capillary *k* allowing for expansion of the liquid.

209. Preparation of Solutions.—A solution of a definite concentration, sufficiently free of color and clear to transmit light, must be prepared. While

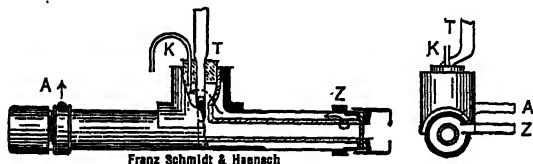


FIG. 73.—Improved Inversion Tube.

solutions of various concentrations may be used, it is advisable to simplify the calculations by using a multiple or convenient fraction of the normal weight dissolved in 100 ml. of solution or a multiple of this volume number.

Dissolve the normal weight (26 grams) of the material contained in a 100-ml. flask, in water and add sufficient subacetate of lead solution, or of the nor-

mal acetate, according to the material to be tested, to precipitate all of the matter precipitable by the reagent. Complete the volume of the solution to the 100 ml. mark of the flask. Mix the contents of the flask thoroughly and pour upon paper for filtration. The entire contents of the flask should be poured upon the paper at once. Reject the first portions of the filtrate or all of it until a perfectly clear solution is obtained. These portions should be used in rinsing the receiving-vessel. The first portions of the filtrate contain the moisture displaced from the filter-paper if the paper is not perfectly dry. With dry filter paper (which should be used for all accurate work) there is an adsorption of moisture<sup>7</sup> and not less than 25 ml. of the filtrate should be discarded when polarizing raw sugars to avoid the effect of this adsorption. If the filtration is slow or the analysis is to be especially accurate, and always in the analysis of sugar, the funnel should be covered during filtration to prevent evaporation. No portion of the filtrate should be poured back through the filter in careful work.

The above description covers the preparation of solutions in a general way. Detailed descriptions will be given in connection with the analysis of the various materials, especially as regards the use of the clarifying agents. The quantity of subacetate of lead to be used in clarifying solutions can be given only approximately on account of the variations in the materials themselves and in the illumination of the polariscope. In general the minimum quantity of the lead-salt that will yield a clear and sufficiently light-colored solution should be used. Excessive use of lead introduces important errors into the analysis. The following numbers refer to cubic centimeters of the lead solution of 54.3° Brix per one normal weight of the material: Raw juice, 0.75 to 2.0 cc.; sirup, 7 to 10 cc.; molasses of 20° Brix (vacuum-pan control), 5 to 10 cc.; first molasses 7 to 10 cc.; final molasses, 25 to 30 cc.; 96° raw sugar, 2 to 4 cc.; 98° raw sugar, 1 to 2 cc.; 80 to 89° second sugar, 5 to 7 cc.; filter press-cake, 6 cc.

Horne's dry subacetate of lead may very often replace the solution of the salt with advantage. (See page 225.) About one-third of a gram of the dry salt is equivalent to 1 cc. of the 54.3 Brix.

Sections 217-219, relative to the influence of the lead-salt upon the sugars and the volume of the precipitates, should be consulted.

Alumina-cream should be used in addition to the subacetate of lead in clarifying the solution in testing a sugar. The alumina-cream alone is usually sufficient in the analysis of high-grade sugars.

It is usually advisable to add a little of the lead reagent to the sugar solution, mix thoroughly, await the subsidence of the precipitate and then test the supernatant liquid with a drop of the reagent to ascertain whether more lead is required. As experienced operator can readily judge by the appearance of the precipitate whether the lead has been used in sufficient quantity. The reagent should be measured in routine work and in so far as is possible the same quantity should always be used with similar materials.

**210. Weighing Dishes.**—The materials in sugar analysis are most conveniently weighed in a nickel or German-silver capsule, made especially for this purpose (shown on scale-pan, Fig. 80). The solutions do not adhere to the

<sup>7</sup> Hardin and Zerban, *The Planter*, Vol. 73 (1924), No. 20.

polished surfaces of the capsule and the shape of this is such that the material may be very readily washed into the flask. The capsules lose weight gradually through use. The counterpoise should not be filed to correct for this loss, but the stem or plug should be unscrewed and sufficient lead be removed from the cavity to compensate.

If the sugar-flask has a narrow neck, as is prescribed in careful work, it requires skill to wash sugar or other material into it. This operation is facilitated by inserting the stem of a small German-silver funnel, made for the purpose, into the neck of the flask. This should reach to just below the neck, thus keeping the sugar from contact with the latter. The solution adhering to the capsule and funnel should be washed into the flask with a jet of water. Sufficient room must be left in the flask—about 20 ml.—to permit giving the liquid a rotary motion, for dissolving the material.

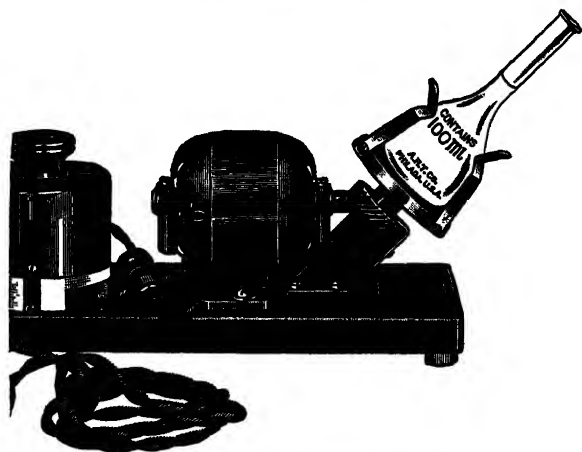


FIG. 74.—Multer Sugar Dissolver.

**211. Dissolving the Material.**—The flask should be held by its neck to prevent the hand from warming the solution. From time to time it should be examined from below to note whether undissolved material remains. Many mechanical devices for dissolving sugars are in use, the one designed by Multer, Fig. 74, being a convenient form. It consists of a motor-driven inclined holder, for a single flask, which rotates slowly, allowing the sugar grains to fall through the solution as the flask turns. In practice it has been found that the device dissolves the normal weight of raw sugar in about the time that an operator takes to weigh out the succeeding sample and wash it into the flask, so one dissolver is ample for the needs of one analyst.

**212. Mixing and Filtering.**—After solution of the material and its clarification with lead subacetate as has been described, the volume should be completed to the mark with water. Should drops of water adhere to the neck of the flask they should be absorbed with strips of filter-paper. A drop of ether

or fusel oil may be poured on the surface of the liquid if foam is present when the solution is close to the mark. In filling to the mark the flask should be held on a level with the eye and the water added drop by drop until the lowest line of the meniscus appears to touch the line on the flask. The water used in preparing the solutions should be either distilled or other water free of optically active substances.

The contents of the flask should be thoroughly mixed by shaking and be poured immediately upon the filter. The filter should be of paper suitable for rapid filtrations, and should be fluted or "star" folded or a ribbed funnel should be used. The funnel and paper should be large enough, especially in analyzing sugars, to receive the entire contents of the flask. The filter-paper should never be so large as to project above the edges of the funnel. The first few cubic centimeters of the filtrate should be used in rinsing the filtering cylinder and should then be rejected. It is always necessary in analyzing sugars and other materials requiring considerable accuracy to reject portions

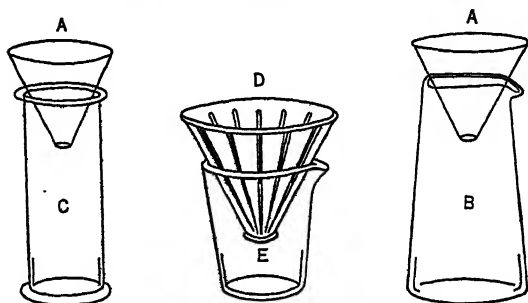


FIG. 75.—Filtering Devices.

of the filtrate until it runs clear. If the filtrate does not clear, it is best to prepare a new solution, changing the quantity of subacetate of lead. The polarization should never be attempted except with perfectly clear solutions.

It may sometimes be difficult to obtain clear solutions with the juice and products of unsound cane. The addition of a little common salt or sodium phosphate followed by refiltration will often remedy this, or both the salt and kieselguhr may be used. Occasionally the difficulty may be due to insufficient subacetate. If so, a little of Horne's dry subacetate should be added.

**Filtering Devices.**—A convenient filtering arrangement is illustrated in Fig. 75. *A* is a stemless funnel, *B* a quarter-pint precipitating-jar, and *C* a small cylinder. A plain cylinder is preferred by many chemists to the lip-cylinder, as the funnel makes a closer joint with the edge. *E* is a "tumbler" beaker of heavy glass which is particularly easy to wash and dry because of its open form. *D* is a copper funnel, fluted, which is convenient and avoids breakage.

Stemless funnels, 4 inches in diameter, made of good tin-plate or of thin copper, planished, are more convenient, except for invert solutions, than glass.

The advantage of the metal stemless funnels and heavy glass precipitating-

jars, cylinders or tumblers is the ease with which they may be washed and dried. The glass vessel serves as a convenient support for the funnels.

**213. Sugar-flasks.**—The flasks used in sugar-work are usually graduated to hold 50 ml., 100 ml., or multiples of 100 ml. They are also graduated with two marks, viz., 50–55 ml., 100–110 ml., etc., and are then called “sugar-flasks” by the dealers.

Orders to dealers for flasks and other precision ware should be very specific in stating the system of graduation, whether to Mohr’s cubic centimeters or units, or metric cubic centimeters (milliliters, ml.). It is important that all such ware in the laboratory be of the same system of graduation. Mohr’s units, used with the old normal weight of 26.048 grams have been largely superseded by the metric or true cubic centimeters at 20° C. (ml.) with the normal weight of 26 grams, adopted by the International Commission for Uniform Methods. It is to be hoped that the older standard will entirely disappear from use.

Flasks should be made from glass tubing of uniform bore and circular cross-section. The shape of the body of the flask should approximate that of the diagrams in Fig. 76. A flask of this form gives little trouble from air-bubbles.

Flasks for commercial work often have necks of larger internal diameter than those used in the U. S. Customs laboratories or in research work. This is unnecessary and the diameters specified below should be adhered to. The following maximum internal diameters of the neck and limits of tolerance of error in the capacity of flasks are specified by the U. S. Bureau of Standards:

Capacity of the Flask, cc. (or ml.)	Internal Diameter of the Neck, mm.	Tolerance of Error, cc. (or ml.)
50	10	0.05
100	12	0.08
200	13	0.10
250	15	0.12
500	18	0.15
1000	20	0.30

This limit of tolerance is too large for flasks of such small neck diameter. Of several hundred flasks purchased of a prominent dealer for the laboratories under the direction of Dr. Spencer and bearing the maker’s certificate of calibration, all were well within the tolerance limits given in the table. A maximum error of .04 ml. should be the requirement for a 100-ml. flask to be used in accurate polarization work. (See further under “Calibration of Sugar Flasks.”)

• The 100 ml. flasks specified for use in the U. S. Custom House laboratories are like *B* of Fig. 76. These have a height of 130 mm. The neck is 70 mm. in length and its internal diameter must be not less than 11.5 mm. and not

more than 12.5 mm. The graduation marks shall be not less than 30 mm. from the upper end and 15 mm. from the lower end of the neck.

The flasks shown in Fig. 76 all conform in shape of the body with the U. S. Customs regulations. They should be distinctly marked with their capacity

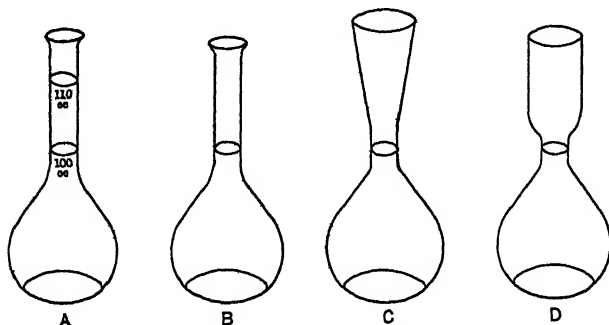


FIG. 76.—Types of Sugar Flasks.

and the system of graduation, *e.g.*, "Contains 100 cc., 17.5/17.5° C." for the Mohr flask and "Contains 100 ml. 20° C." for the metric or true cubic-centimeter flasks. The graduation mark should completely encircle the neck of the flask.

Pellet's conical flasks, Fig. 77, are of strong glass and have a rubber cover to slip over the bottom and a ring for the neck to reduce breakage. Their form gives them great stability and facilitates the escape of air-bubbles.

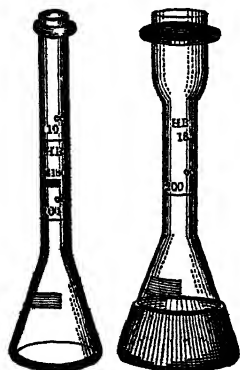


FIG. 77.—Pellet's Conical Flasks.

Referring to Fig. 76, the flasks *A* and *B* of various capacities fill most of the requirements of the sugar-house laboratory. The *Stift* (*C*) and *Kohlrausch* (*D*) flasks are used in the analysis of filter press-cake. The flask *C* if narrow at the graduation may be used in all classes of work.

The flasks should be frequently and thoroughly cleaned. C. A. Browne<sup>2</sup> recommends cleaning with a warm solution of sodium hydroxide and Rochelle salts, such as is used in preparing Fehling's solution. This removes the film of lead carbonate that deposits upon the walls of the flask. Strong commercial muriatic acid is usually used for this cleaning in sugar-house laboratories. Treatment with nitric acid, followed by washing and then a strong solution of chromic acid in concentrated sulphuric acid, is good preparation of flasks for calibration. It is advisable to use the chromic acid solution frequently in

<sup>2</sup> "Handbook of Sugar Analysis," p. 171.

cleansing flasks. After this treatment the drops of water will drain from the neck of the flask properly instead of adhering to it.

*Calibration of Sugar-flasks.*—No flask should be used in important work without having first verified its marked capacity. There is much confusion on the part of manufacturers between the true and Mohr's cubic centimeter, flasks of the one system being sometimes marked as having been graduated to the other.

Cleanse the flask as has been described above and thoroughly dry it in an oven. On cooling the flask note whether moisture has condensed upon the inside walls and if so return the flask to the oven.

The weighing should be by substitution to eliminate errors of the balance itself. Cool the flask to room temperature in the balance case, but do not wipe it again, and then accurately counterpoise it, placing it upon the left-hand balance-pan. Pieces of metal or weights may be used in counterpoising. Remove the flask from the balance and counterpoise the weight on the right-hand pan with accurate weights substituted for the flask. This gives the weight of the flask to the limits of accuracy of the analytical weights. Fill the flask to the mark with recently boiled distilled water of room temperature. A large pipette should be used in running the water into the flask to avoid so far as is possible wetting the neck. Remove water that may adhere to the neck of the flask, with a roll of filter paper. Verify the filling of the flask by holding it by the upper part of the neck with the graduation at the level of the eye. If the lower part of the curve of the meniscus is not in line with the graduation mark, add or remove water by means of a small pipette and bring it into line.

Place the filled flask upon the balance-pan as before and counterpoise it with pieces of metal or weights. Remove the flask and note the temperature of the water with an accurate thermometer. Counterpoise the metal with the analytical weights and record this weight as that of the flask and water. Deducting the weight of the flask gives the correct weight of the water whether the balance is in proper adjustment or not.

Reference is now had to the tables, pages 433 and 431, showing the weight of 100 cc. (according to Mohr or to the true cubic centimeter table) at various temperatures. For example: Our flask contains 99.958 grams of water at 20° C. Reference to the table shows this to be the apparent weight of 100 Mohr's cubic centimeter at 20° C., hence the flask is correctly graduated to this system. Similarly using the same flask and weight of water and the table of corrections for true cubic centimeters at 20° C. The correction at 20° C. is +0.282, which added to 99.958 gives 100.240 cc. as the true capacity of the flask. For comparison only, subtracting .006, the cubical expansion of the Mohr's flask from 17.5° to 20°, we have  $100.240 - .006 = 100.234$  cc. These numbers show the actual relation between flasks graduated to true and Mohr cubic centimeter, viz., 100 : 100.234.

**214. Spencer Flask Calibrating Device.**—This instrument was designed by Dr. Spencer to permit of accurate graduation of flasks at the Central Control Laboratory of the Cuban-American Sugar Company at Cardenas, Cuba. Primarily, it is a method of recording by electrical contact the exact

surface of the water in a flask and at the same time marking this surface-level accurately on the outside of the flask. The drawing, Fig. 78, shows the construction of the device and a description of the method of calibrating a flask will make the operation plain. Flasks of the right form and size required to hold the desired volume are purchased in "blank" with no graduation mark. In addition to the calibrating device a series of Morse Calibrating Burettes (shown at A, Fig. 78) covering the volumes of flasks to be marked are required.

These burettes should be standardized by the Bureau of Standards at Washington.

The first step is to adjust the graver-point, *P*, and the platinum electrodes, *E*, so that they are exactly opposite each other. This is done, with no flask in the instrument, by loosening the lock-nut *L* and raising or lowering the electrode-carrier *C* at the same time that the nut *N* holding graver *G* is loosened. The graver is pushed forward until it just touches the higher of the two points of the electrodes. The adjustment need be made only once for a series of flasks. The lock-nut, *L*, is tightened, and the graver

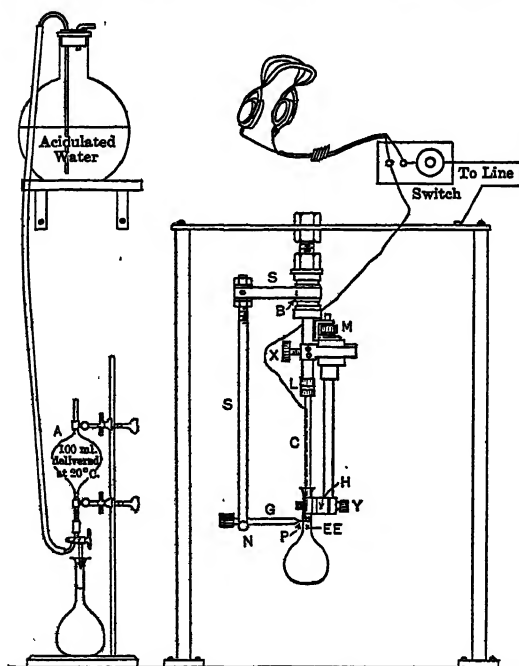


FIG. 78.—Calibrating Device (Spencer).

is drawn back, after which the flask-holder *H* is lowered as far as it will go by turning the milled head, *M*, and the flask is put in place for centering only. By means of the screws *X* and *Y* the adjustment of the flask is made so that the electrode holder will be approximately in the middle of the neck of the flask when the flask is raised.

The flask is now removed from the holder without disturbing this adjustment and the outside of the neck is coated with a film of molten beeswax and rosin which is allowed to cool. The flask, which has been dried previously, is now ready to be filled. From the proper Morse burette the exact volume of acidulated water ( $H_2SO_4$ —1 : 1000) is run into the flask, which is promptly placed in the flask holder of the calibrating device and raised with the milled head until the electrode points approach the surface of the water. The



current is now turned on, the head phones are adjusted to the ears, and the flask is raised very cautiously by means of the milled head until contact is made by the platinum points touching the surface of the water. This is apparent by the hiss in the earphones. It is now evident that the surface of the liquid in the flask is exactly on a level with the graver-point on the outside. The graver is moved forward until the point penetrates the wax. (There is a spring behind the point which permits of definite contact with the glass and takes up any minor inequalities in the shape of the neck.) The whole marking sweep, *S*, is then swung completely around the flask on the bearing, *B*, so that the wax is cut away by the sharp point in a complete circle exactly at the liquid level. By means of "Diamond Ink" or some similar hydrofluoric acid preparation the flask is etched at this circle in the wax, after the flask has been removed from the instrument.

It is obvious that the work must be done in a place free from drafts and vibration. Between the time that the flask is filled and the mark made on the wax care should be taken to prevent any change in temperature.

Flasks marked in this manner and rechecked by the weighing method given in the previous section were found to be extremely accurate; over 93 per cent of the flasks so checked showed an error of less than .025 ml., while not one had an error amounting to .05 ml. The Bureau of Standards has adopted the Spencer device with certain modifications to suit its purposes.

**215. Balances. Wash-bottles. Stock-bottles. Heating Devices.**—*Balances.*—Convenient types of balances for sugar work are shown in Figs. 79, 80, 81 and 82. In addition a good analytical balance and a set of high-grade weights are required. The analytical balance is used in ash determinations, glucose tests, calibration of sugar flasks and in the many special investigations. A "sugar" balance of good accuracy and a capacity of about 300 grams is needed in weighing samples for polarizations. For use in tropical and subtropical countries this balance should be fitted with agate bearing and knife edges. The balance shown in Fig. 79 is suitable.

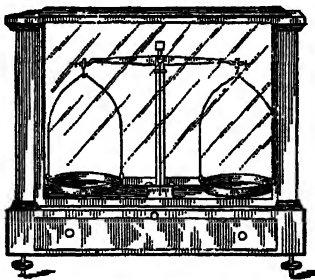


FIG. 79.—Sugar Balance.

The Bates balance, Fig. 80, especially designed for polarization work, has several convenient features. The customary bow has been replaced by a single bow at the back, giving free access to the pans and reducing spilling of sugar to a minimum. The "scoop" or weighing-dish balances either of the pans and is interchangeable with them, thereby avoiding the use of a counterpoise weight. Two sets of sugar weights, normal and half normal, should be provided. One set of these weights should be kept in reserve for verifying and checking the weight of the others. The balances should at least be sensitive to 2 mg. with a full load in the pans, though weights of sugar materials for polarization, to within 2 mg. are usually sufficiently accurate. The errors

that may be introduced through evaporation or absorption of moisture in slow weighing are of more importance than weights to within 2 mg.

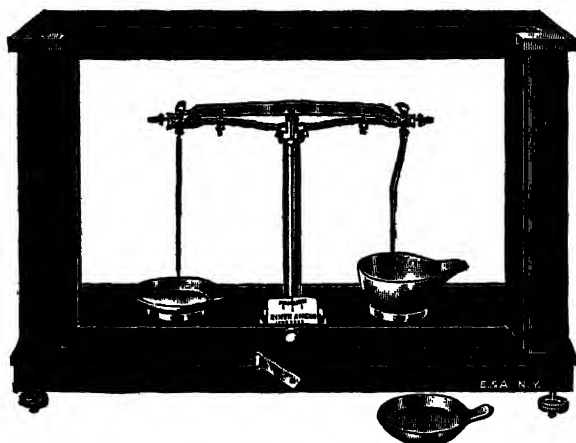


FIG. 80.—Bates Sugar Balance.

The bullion type of balance shown in Fig. 81 is very convenient for the so-called rough weighings in which large capacity and speed of manipulation are essentials. This balance should be placed in a glass-framed hood to protect it from dust and currents of air. This instrument is made in several capacities.

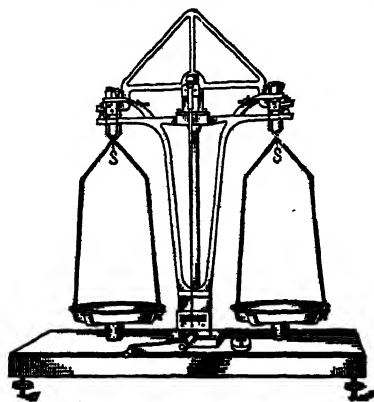


FIG. 81.—Balance for Rough Weighings.

That of 5 kg., sensitive to 100 mg., is a suitable size. It should have agate bearings and knife edges for tropical work. This scale is suitable for use in bagasse analysis and in determining the degree Brix of massecuites and molasses. An ordinary "trip" balance is also useful in this type of work (Fig. 82).

*Wash-bottles. Stock-bottles.*—The water and the lead solution for use in sugar analysis should be kept in large bottles on a shelf above the work-table. The water-bottle should have a glass syphon-tube with rubber connections, a pinch-cock and glass-nozzle, forming a convenient arrangement for washing samples into the sugar-flask and for diluting the solutions to the graduations on the necks of the flasks. This is a very satisfactory form of wash-bottle, and several of these should be distributed about the laboratory. A double

nozzle connection, one having a glass tip of large size and the other a fine glass tip is an added convenience, the first to permit of rapid filling and the second of fine adjustments to the mark of flasks, etc. The lead subacetate bottle should be connected with a reservoir burette. The burette has a three-way cock, one opening connecting with the stock-bottle by means of a glass syphon and a rubber tube. The air-inlet to the subacetate-of-lead bottle should be provided with a small wash-bottle containing caustic soda solution, to absorb the carbonic acid and prevent precipitation of the lead. This is not strictly necessary, since a small precipitation is not objectionable, but where very large stock-bottles are used the air-washing arrangement should be employed.

Where lead subacetate solution is used in storing samples of juice a stock-bottle containing the concentrated solution should also be provided.

*Heating Devices.* — The electric hot-plate is the most convenient heating device for laboratories having a twenty-four-hour electric service. Where the generators are only operated at night the hot-plates must be supplemented by stoves. Various types of alcohol and gasoline stoves are on the market that are satisfactory for heating in inversions, extractions, etc.

216. Notes on Polariscopic Manipulations. — The screw-caps of the observation-tubes should not bear heavily upon the cover-glasses, since glass is double-refracting under these conditions and does not quickly recover its normal condition. A large error may be introduced through excessive pressure on the glasses. The cover-glasses should be of the best quality of glass, perfectly clean and with parallel sides. A glass may be tested with regard to the parallelism of its surfaces by holding it in front of a window and looking through it at a window-bar; on revolving the glass slowly between the thumb and a finger, if the bar appears to move the surfaces are not parallel and the glass should be rejected. Old glasses which have become slightly scratched should not be used. Glasses and observation-tubes should be frequently washed with acetic acid.

The planes of the ends of the observation-tubes should be perpendicular to the axis of the tube. This may be tested by placing a tube filled with a sugar solution in the trough of the polariscope and making an observation; on revolving the tube in the trough, and making observations at different positions, should the readings vary, the ends of the tube have not been properly ground.

The manufacturers of polariscopes and their accessories have attained such accuracy in their methods that faulty apparatus rarely leaves their workshops, nevertheless the scales and accessories should be checked to verify graduations and tube length.

The polariscope should be used in a well-ventilated room. It should be

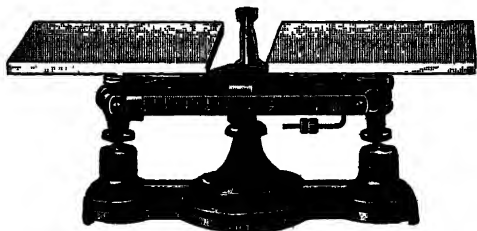


FIG. 82.—Trip Balance.



disturbing influence, should increase the polarization more than is due to the sugar, since the volume of the solution is decreased.

Letting  $x$  = the weight of molasses, and  $y$  = the polariscopic reading, the ratio  $\frac{y}{x}$  should increase with the concentration, if there is an error, due to the volume of the precipitate, not compensated by other influences. Sachs used quantities of beet-molasses ranging from 5 to 35 grams in 100 cc. and substituting the values of  $x$  and  $y$  in the ratio and reducing he obtained the following figures:

1st Series.....	1.906	1.900	1.900	1.906	1.896
2d Series.....	2.14	2.13	2.14	2.14	

The practically constant value of the ratio shows that minus errors have fully compensated for that due to the volume of the precipitate.

In a similar experiment with beet-juices Sachs obtained the following numbers:

1st Series.....	0.5446	0.5474	0.5480	0.5497
2d Series.....	0.5800	0.5830	0.5842	0.5860

The increase in the ratio with juices shows that there is an error due to the volume of the precipitate and there is not sufficient compensating error to correct it.

Experiments by Spencer in Cuba with cane-molasses gave results similar to those of Sachs, and several series of unpublished analyses of cane-juices made by L. R. Cook, at his instance, gave the following results:

Juice and Lead.	25 Gr.: 1 cc.	50 Gr.: 2 cc.	75 Gr.: 3 cc.	100 Gr.: 4 cc.
1st Series.....	0.628	0.628	0.632	0.633
2d Series.....	0.628	0.630	0.633	0.634

Series No. 2 is a duplicate of No. 1. The polarization of the juice using the normal weight in 100 cc. was 16.36, and that using four times the normal weight in 100 cc. and dividing the polariscope reading by 4 was 16.49, showing an uncompensated error of 0.13 per cent of sucrose. The uncompensated error in the 2d Series, using the same juice, was 0.15.

In two other series of experiments by Cook, in the first of which the polarization was immediate and in the second after forty hours, the results were as follows:

Juice and Lead...	20 Gr.: 1 cc.	40 Gr.: 2 cc.	60 Gr.: 3 cc.	80 Gr.: 4 cc.	100 Gr.: 5 cc.
1st Series.	0.690	0.691	0.690(?)	0.694	0.696
2d Series.	0.687	0.690	0.692	0.693	0.694

As in the case of Sachs's experiments with beets, this work of Cook shows that there is a very perceptible error in the analysis of cane-juices due to the volume of the precipitate.

Sachs<sup>11</sup> used the following method of determining the volume of the precipitate: Clarify 100 cc. of juice with subacetate of lead as usual, using a tall cylinder instead of a flask. Wash the precipitate by decantation, first with cold water and finally with hot water, until all of the sucrose is removed. Transfer the precipitate to a 100-cc. flask and add one-half the normal weight of cane-sugar, dissolve the sugar, and dilute the solution to 100 cc., mix, filter, and polarize, using a 400-mm. observation-tube. The results are calculated as follows:

Let  $P$  = per cent of sucrose in the sugar;

$P'$  = the polarization of the solution, made up in the presence of the precipitate;

$x$  = the volume of the lead precipitate.

Then

$$x = \frac{100P' - 100P}{P'}$$

Example: Let  $P = 99.9$ ;

$$P' = 100.77.$$

Then

$$x = \frac{(100 \times 100.77) - (100 \times 99.9)}{100.77} = 0.86 \text{ cc.}$$

The following is the method of Scheibler<sup>12</sup> for correcting for the error due to the volume of the precipitate, and usually termed "Scheibler's double-dilution method": To 100 cc. of the juice add the requisite quantity of subacetate of lead for the clarification, complete the volume to 110 cc. and polarize as usual; to a second portion of 100 cc. of juice add lead as before, complete the volume to 220 cc., and polarize.

Calculation: Multiply the second polariscope reading by 2, subtract the product from the first reading, multiply the remainder by 2.2 and deduct this product from the first reading. The remainder is the required reading for the calculation of the per cent sucrose:

#### Example

Degree Brix of the juices.....	18.0
First polariscope reading (110 cc.).....	57.6
Sound polariscope reading (220 cc.).....	28.7

<sup>11</sup> *Loc. cit.*

<sup>12</sup> *Zeit. Rübensucker-Industrie*, 25, 1054.

$2 \times 28.7 = 57.4$ ;  $57.6 - 57.4 = 0.2$ ;  $2.2 \times 0.2 = 0.44$ ;  $57.6 - 0.44 = 57.16$   
= corrected polariscope reading. By Schmitz table, page 481 we have:

15.18

.03

.02

---

15.23 = required per cent.

The calculations as given here are modified for application to juices.

In applying this method to other sugar-house products, using the normal weight, calculate as follows: First volume 100 cc.; second volume 200 cc.; multiply the second polariscope reading by 2 and subtract the product from the first reading; multiply the remainder by 2 and subtract the product from the first reading. This last remainder is the per cent of sucrose.

It is evident that this method demands extreme care in making the tests, since an error is multiplied.

**218. Horne's "Dry Lead" Method.**—W. D. Horne<sup>13</sup> recommended the use of dry subacetate of lead to eliminate the volume of precipitate error. The finely powdered lead salt is added to the solution after dilution to volume. Thorough mixing and filtration follow. If the lead salt has been added in just sufficient quantity for the clarification, there is no dilution of the solution and no precipitate error. All the precautions necessary in the use of the lead in solution as regards precipitation of reducing sugar, influence upon specific rotation, etc., must be observed with the dry-salt.

This "dry-lead" method has never been adopted for raw sugar polarizations either by commercial or factory laboratories or by the Government, although it is authorized by the Official Methods of the Association of Official Agricultural Chemists as an alternative of the usual method with sub-acetate of lead solution. It has proved of great value in sugar analysis, however, and is almost universally used in routine "purity" determinations and other control tests in beet-houses, cane-factories and refineries. Rapidity and accuracy are gained by its use in these tests since the necessity of a dilution factor is avoided. The anhydrous lead sub-acetate employed should be only that especially prepared for sugar analysis and should contain 72.8 per cent of lead. One-third of a gram of the salt is equal to 1 ml. of the regular 54° Brix lead solution. The dry lead is also very valuable as a preservative in the storage of juice samples and its use for this purpose is general. Extensive experiments by Dr. Spencer have proved that raw juices may be stored in hot climates for a period of several days without appreciable deterioration using no more of the lead salt than is necessary for the clarification of the juice. (See p. 302.)

**219. Influence of Subacetate of Lead and other Substances upon the Sugar and Optically Active Non-sugars in Cane Products.**—*Sucrose.*—The rotatory power is not modified by subacetate of lead under the usual analytical conditions. In the presence of a very large excess of the lead salt, there is a slight

<sup>13</sup>Jour. Am. Chem. Soc., Vol. 26, p. 186.

diminution of the rotatory power; there is a decided diminution in alcoholic solution containing the lead salt.

The investigations of Bates and Blake, U. S. Bureau of Standards,<sup>14</sup> show that errors of importance in rotation are caused by excessive amounts of subacetate of lead. An excess of 0.5 cc. causes a diminution of  $0.1^\circ$ ; 1 cc. of  $0.12^\circ$ ; 2 cc. of  $0.11^\circ$  and 3 cc. a diminution of  $0.90^\circ$  on the cane-sugar scale. The rotation reaches a minimum value when an excess of 1 cc. is present and returns to the initial value with 6 cc. in excess and continues to increase with the amount of lead solution added. A sugar polarizing  $99.9^\circ$  was used in these experiments.

Farnsteiner<sup>15</sup> made the following observations relative to the influence of certain inorganic salts:

With constant relation of sugar to water, the chlorides of barium, strontium, and calcium cause a decrease in the rotation, which continues to decrease as the salt is increased; calcium chloride causes a decrease, but when the salt reaches a maximum further addition causes an increase which finally exceeds that of the pure-sugar solution.

If the relation of the sugar to that of the salt be kept constant, it is found that the addition of water causes in all cases an increase in the specific rotatory power, i.e., the action of the salts is lessened. The specific rotatory power is almost unaffected by varying the quantity of sugar with a constant relation between the salt and water. The chlorides of lithium, sodium, and potassium behave in a similar manner.

An examination of the action of the same quantities of different salts shows that in the case of strontium, calcium, and magnesium the depression varies inversely with the molecular weight, and that the product of the two quantities is approximately a constant. Barium chloride does not act in the same manner, but the chlorides of the alkalis show a similar relation. The relation, however, only holds good within each group of chlorides and not for two salts belonging to different groups.

The rotatory power of sucrose in water or in alcohol solution is not modified by the presence of nitrates of sodium and potassium even when the quantity of the nitrate amounts to as much as 50 per cent of the sucrose (E. Gravier).

In his investigation of the influence of the lead precipitate, Sachs<sup>16</sup> found that the presence of acetate of potassium very perceptibly diminished the rotation. The diminution was also noticeable with the sulphates of potassium and lead, but was not so marked with the corresponding sodium salts. Sachs also states that citrate of potassium, carbonate of sodium, and several other salts have an influence analogous to that of the acetates. The presence of free acetic acid reduces this influence in part. Sachs further states that the use of tannic acid in decolorizing solutions is very objectionable on account of the volume of the precipitate formed with the lead.

*Dextrose*.—The rotatory power of dextrose is not modified, or, if at all, but very slightly, by either the subacetate or neutral acetate of lead, under the analytical conditions. See also Invert-sugar.

*Levulose*.—The rotatory power of levulose is very greatly diminished by the presence of subacetate of lead. Acetic acid added to acidity restores the rota-

<sup>14</sup> Bulletin No. 3, p. 105.

<sup>15</sup> Berichte deut. chem. Gesell. 23, 3570; Journ. Chem. Soc., 60, 283.

<sup>16</sup> Revue Universelle de la Fabrication du Sucre, 1, 151.



tion of the levulose (Gill in 1871, Spencer in 1885, Pellet). Levulose is partly precipitated as a lead salt in the presence of certain chlorides, in quantities greater or less, according to the relative proportions of the salts, lead, and levulose (Pellet, Edson). Basic acetate of lead precipitates levulose in part, when salts occur in the same solution with constituents of which basic acetate of lead forms insoluble combinations (Prinsen-Geerligs).

*Invert-sugar, Dextrose, and Levulose.*—In the presence of the salts formed in the decomposition of the subacetate of lead, dextrose, and levulose are precipitated in part (Pellet, Edson). The influence of the basic lead salt on the rotatory power of levulose (see Levulose) or the formation of levulosate of lead of little optical activity gives undue prominence to the dextrose, which is not affected and results in a plus error. Increasing amounts of subacetate of lead added to invert-sugar solutions decrease the left rotation, and finally the rotation is to the right on account of that of the dextrose. C. H. Gill called attention to this error in the Journal of the Chemical Society, April, 1871, and in the early editions of this book, Spencer advised the use of acetic acid to restore the rotatory power of the levulose.

Acetic acid slightly lowers the rotatory power of invert-sugar; hydrochloric acid has an opposite effect. Sodium acetate and sodium chloride increase the rotation (H. A. Weber and Wm. McPhersons). Sulphuric and hydrochloric acids increase the rotation; oxalic acid has no effect. The rotation increases as the quantity of the mineral acid is increased. If the invert-sugar solution be diluted after warming with hydrochloric acid, it does not quickly reach the rotation corresponding to its dilution, which requires about twenty-four hours (Gube <sup>17</sup>).

*Malic acid.*—This acid is lævorotatory. The artificial malic acid is optically inactive. Malic acid is precipitated by subacetate of lead.

*Pectine and Parapectine.*—These substances are dextrorotatory and are both precipitated by subacetate of lead, and the second by normal acetate of lead.

*Asparagine.*—Not precipitable by subacetate of lead, but is rendered dextrorotatory instead of lævorotatory by the lead salt. In water solution and alkaline solution asparagine is lævorotatory; in acid solution, dextrorotatory.<sup>18</sup> Asparagine is insoluble in alcohol, and in the presence of acetic acid is inactive.<sup>19</sup> In neutral and alkaline solution asparagine is lævorotatory; in presence of a mineral acid, dextrorotatory; in the presence of acetic acid the rotation is diminished and with 10 molecules of the acid becomes 0°, and with additional acid is dextrorotatory (Degener). Asparagine is present in cane-juice, especially that from immature cane; it is changed to aspartic acid by the action of lime, and as a lime salt is found in molasses.<sup>20</sup>

*Aspartic Acid.*—From asparagine by the action of lime; the lime salt is soluble. In alkaline solutions, aspartates are lævorotatory and in acid solutions dextrorotatory. Aspartic acid is precipitated by subacetate of lead.

<sup>17</sup> Bulletin Assoc. Chimistes de France, 3, 131.

<sup>18</sup> Optical Rotation of Organic Substances, Landolt—Dr. Long's Eng. ed. 541.

<sup>19</sup> Champion and Pellet, *Compt. Rend.*, 82, 819.

<sup>20</sup> W. Maxwell, Bul. 38, 2d Series, La. Expt. Station, p. 1380.

**220. Boneblack Error.**—Boneblack or animal charcoal exercises an adsorbent action on sugars and the impurities present in sugar products. For this reason its use in analytical work has been discontinued and most official methods definitely exclude its employment. Modern high-power electric lights in polarizing have made it possible to dispense with it.

**221. Influence of Temperature upon Polarizations.** *Corrections for Temperature.*—The compensating type of polariscope can only give correct readings at the temperature at which it was standardized. The former standard temperature for these instruments was 17.5° C. and all the instruments made prior to 1898 conformed to this standard. In 1897 the International Commission for Uniform Methods of Sugar Analysis adopted 26 grams as the normal weight and 100 true cubic centimeters at 20° C. as the corresponding volume. The instrument makers have since conformed with this specification.

The rotation due to the quartz wedges increases with rise of temperature and that of sucrose decreases giving a combined effect of a decrease in the reading of a normal weight of pure sucrose of 0.03° Ventzke for each 1° C. increase in temperature. The United States Treasury Department were the first to use temperature corrections in polarizations, these being based on tables prepared by Dr. H. W. Wiley.

C. A. Browne<sup>21</sup> made a full study of the influence of temperature in the polarization of raw sugar, and stated that the formula

$$P^{20} = P^t[1 + .0003(t - 20)]$$

in which  $P^{20}$  is the corrected polarization,  $P^t$  the observed polarization and  $t$  the temperature of the observation, may be applied without serious error to cane-sugars polarizing above 96°. (In actual practice, many laboratories simplify the calculation by merely adding .03°  $V$  for each degree above 20° C. The error introduced by this method is slight but the use of the equation is to be recommended.)

Dr. Browne concluded that the only strictly accurate method was to polarize at 20° C. and he equipped the New York Sugar Trade Laboratory (of which he was then in charge) with a constant temperature room in which all sugar solutions are prepared and polarized at 20° C. The Treasury Department has since installed similar constant temperature laboratories in some of the larger ports for use in polarizing sugars for duty purposes. The temperature correction formula as given above is used by Government laboratories not so equipped. Few industrial sugar laboratories could justify the expense of standard temperature rooms, particularly in the tropics. The practical accuracy of the correction formula has been demonstrated year after year by comparisons of refinery control polarizations corrected by the formula, with polarizations of the same sugars in the New York Sugar Trade Laboratory where the standard temperature room at 20° is employed; average polarizations by the two methods always being extremely close.

The formula is based on the effect of temperature on the rotation of pure

<sup>21</sup> Handbook of Sugar Analysis. N. Y., 1912, pp. 255-262.

*sucrose*, and as the impurities increase (particularly levulose) the formula loses its validity. Browne has shown that at about 80° Ventzke the effect of temperature upon the rotation of levulose and other impurities counterbalances the decrease in the rotation of the sucrose and quartz wedges, so that at this point no temperature correction at all is applied. Below 80° V. the temperature coefficient of sucrose is generally more than overbalanced so that the polarization *increases* with a rise in temperature. The following formula is applicable to cane products other than raw sugars of 96° or above:

$$P^{20} = P^t + 0.015(P - 80)(t - 20).$$

It is to be understood that this formula is for materials of average composition, and will fail to give accurate results in individual cases where the composition is unusual, particularly as to levulose content.

Obviously certain precautions can, and should, be observed to reduce the effect of temperature. The laboratory and polariscope room should be well ventilated and of the same temperature. The polariscope should not be in the vicinity of a heated wall or the laboratory-ovens. The solutions should be prepared and polarized at room temperature. Composite samples of sugar, used in part as a basis of the technical reports (run reports), should be polarized at a time of low temperature in tropical laboratories rather than in the heat of the afternoon.

**222. Limits of Accuracy in Saccharimetric Analysis.**<sup>22</sup>—Dr. C. A. Browne notes twelve errors that may enter into sugar analysis. While these refer specifically to sugar-testing they apply also to a considerable extent in all other analyses:

1. Loss of moisture during mixing.
2. Loss of moisture during weighing.
3. Error in normal weights.
4. Volume of precipitate in clarification.
5. Precipitation of levulose.
6. Error in capacity of flasks.
7. Imperfect mixing of contents of flask.
8. Evaporation during filtering.
9. Error in length of polariscope tubes.
10. Omission of bichromate cell.
11. Variations in temperature.
12. Defects in scales of saccharimeters.

The errors 3, 6, 9 and 12 are in general mutually compensating and with careful management do not appear in the results. Since flasks and tubes may be selected that vary but slightly on each side of the correct numbers, the errors disappear in the averages of duplicate tests. The weights may easily be kept

<sup>22</sup> Dr. C. A. Browne read a paper with this title before the New Orleans Section of the Am. Chem. Soc., Nov. 20, 1914. The comments and conclusions in this paragraph are abstracted from this paper, including the summing up of the residual error.

within the limits of accuracy of the balance. The scales of the polariscope may be checked near the important points with accurate standard quartz plates and corrections be made. Other points and in fact all parts of the scale may be checked with the Schmidt & Haensch control-tube, Fig. 64. Dr. Browne estimates that the following plus errors may enter in careless work. These are numbered as in the previous list:

Error Due to:	Sugar Degree
1. Mixing on paper.....	+0.05
2. Evaporation during weighing.....	+0.02
4. Volume of precipitate in clarification.....	+0.18
5. Precipitation of levulose.....	+0.03
7. Imperfect mixing of the contents of the flask.....	+0.05
8. Evaporation during filtering.....	+0.04
10. Omission of the bichromate cell.....	+0.07
11. Temperature variation from the standard.....	+0.04
	<hr/>
Total error.....	+0.48

The last four errors amounting to  $0.20^\circ$  are preventable and the first four may be reduced. The final residual error in careful work should not exceed  $+0.12$  as follows:

Error Due to:	Sugar Degree
1. Evaporation in mixing.....	+0.010
2. Evaporation in weighing.....	+0.005
4. Volume of precipitate.....	+0.090
5. Precipitation of levulose.....	+0.015
	<hr/>
Total error.....	+0.120

There are other sources of error that have already been considered in the previous pages of this work, such as those introduced by pressure upon the cover-glasses, pressure upon or the wiping of the standardized quartz-plates, preparation of the solution at a different temperature from that of the observation, etc. Such errors are entirely avoidable.

The use of alumina cream or the normal acetate of lead in the clarification instead of the basic acetate, when the material will permit, practically eliminates the levulose error. Horne's dry-lead method removes, or nearly so, the volume of precipitate error. It is possible that both these errors would be reduced by the use of dried acetate of lead in clarifying, where conditions of color will admit.

The temperature error is one of the most important with which the tropical chemist must deal. This may amount to  $-0.5^\circ$  in extreme cases. Since a constant temperature laboratory is an improbable addition to a sugar-house, the only defense against this error lies in making important tests early in the morning during the low-temperature period, and in the use of the temperature correction formulæ given in 221.

## CHAPTER XVIII

### CLERGET SUCROSE. DOUBLE-POLARIZATION METHODS

**223. Clerget's Method for Sucrose.**—This is often termed the "double-polarization method," since two polarizations are made, one before and one after inversion, in order to eliminate the influence of other optically active sugars that may be present with the sucrose. Cane-sugar products usually contain the three sugars, sucrose (+), dextrose (+) and levulose (-). The direct polarization is therefore the resultant of the polarizations of these three sugars. The principle of the Clerget method is to supplement this direct polarization with a second polarization after complete inversion of the sucrose by suitable means. The rotation of the sugars other than sucrose remains constant and the change in polariscopic reading before and after the inversion is due to the inversion of the sucrose. The sucrose is calculated by dividing this change in the polariscopic reading, due to inversion, by the Clerget constant; this constant representing the algebraic difference in the polariscopic readings of pure sucrose, before and after inversion, under the conditions of the method used. Suitable correction is made for the temperature at which the polariscope is read. In theory the Clerget is simple but practically it requires the most exacting and rigorous adherence to correct technique, and the many modifications and changes which are constantly advocated are evidence of the troublesomeness of the method. Possibly no other phase of sugar analysis has been the subject of so much research.

The inverting agents employed are invertase and hydrochloric acid, the invertase method being the more exact but having many attendant difficulties in the preparation of the substance and the time required for the inversion. Dry invertase is now prepared commercially<sup>1</sup> and has been found to keep well even in the tropics. The acid inversion is generally used in factory laboratories. It is recognized that, in common with most sugar analytical methods, the results are more or less close approximations chiefly of value from a comparative standpoint. The modifications of the acid-inversion have been devised with a view to simplifying the work or reducing the error through decomposition of levulose.

**224. Clerget's Method. Herzfeld's Modification.**<sup>2</sup>—Dissolve 52 grams of substance in water in a 200 ml. flask; add subacetate of lead for clarification. then 1-2 ml. alumina cream, dilute to mark, mix well and filter. (In the case of low-grade massecuites or molasses or other dark-colored material weigh 26 grams to 200 ml. or 300 ml., calculating results to the basis of the normal weight per 100 ml.) Delead by adding powdered sodium oxalate, a little at a

<sup>1</sup> Wallerstein Laboratories, 171 Madison Ave., N. Y. City.

<sup>2</sup> Adapted from Official Methods, A. O. A. C.

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time, avoiding an excess. Filter again using dry kieselguhr to aid filtration. Add 50 ml. of this lead-free filtrate to a 100 ml. flask, make to mark, mix well and polarize in a 200 mm. tube. The result times 2 is the direct reading ( $P$ ).

To a second 50 ml. portion in a 100 ml. flask add 25 ml. of water; then little by little, while rotating the flask, 10 ml. of hydrochloric acid 1.1029 sp. gr.  $20^{\circ}/4^{\circ}$  C. ( $24.85^{\circ}$  Brix at  $20^{\circ}$  C.). Heat a water-bath to  $70^{\circ}$  C., place the flask, with the thermometer inserted, in the bath and agitate constantly until the contents of the flask reach  $67^{\circ}$  C., which should require from  $2\frac{1}{2}$  to 3 minutes. Then continue the heating of the solution for exactly five minutes from the moment the thermometer reaches  $67^{\circ}$ , during which time the temperature of the contents of the flask should rise to about  $69^{\circ}$  C. At the end of the five minutes plunge the flask into cold water and cool to about room temperature.

Wash the solution from the thermometer into the flask and complete the volume to 100 ml. at the temperature of the direct polarization. If the invert-sugar solution is too dark-colored it should be treated with successive small portions of metallic zinc-dust after completion of the volume. The coloring-matter is destroyed by the nascent hydrogen. No more zinc than is necessary should be used.

Make the invert polarization, using a Landolt inversion-tube, Fig. 71. Circulate water of the temperature of the direct polarization through the water-jacket of the tube during the operation. Note the polariscope-reading (minus) and ascertain the temperature by inserting an accurate thermometer, graduated to  $\frac{1}{2}^{\circ}$  C., into the solution through the side tubule. Reduce the polariscope-reading to terms of a normal solution observed in a 200-mm. tube. (In the case as here given, reading  $\times 2$  = invert reading  $I$ .) Calculate the sucrose by the following formula:

$$S = \frac{100(P - I)}{143 + 0.0676(m - 13) - T/2'}$$

in which

$S$  = sucrose per cent;

$P$  = direct reading calculated to a basis of normal solution;

$I$  = invert reading calculated to a basis of normal solution;

$T$  = temperature at which readings are made;

$M$  = grams of solids in 100 ml. of invert solution as read (solids by refractometer multiplied by sp. gr. of solution).

It will be noted that in this formula  $m$  denotes total solids and not sucrose because it has been found by Zerban and others that *water* concentration and not sucrose concentration regulates the Clerget divisor. For routine laboratory procedure the solids will be known with sufficient exactness from the composition of the material, if a refractometer is not at hand.

The inversion may be conducted at the room temperature. The inversion is always complete within twenty-four hours at a temperature above  $20^{\circ}$  C. and when there is certainty that the temperature is above  $25^{\circ}$  ten hours is sufficient. The twenty-four-hour period is usually the more convenient. From this it appears that, when time is available, the tedious inversion with heating may be avoided with the avoidance of destruction of levulose.

When inversion is at room temperatures the calculation is as above except that 143.2 is the Clerget constant instead of 143.

**225. Clerget's Method as Modified by Steuerwald.**<sup>2</sup>—The inversion is conducted at room temperature with increased acid strength. A special table of constants is required.

Prepare the solution as described in the preceding paragraph. Measure 50 ml. of the filtrate into a 100 ml. flask and add 30 ml. of hydrochloric acid of 1.1 sp. gr. (acid of 1.188 sp. gr. diluted with an equal volume of water). Set aside three hours if the temperature is between 20° and 25° C. or two hours if above 25° C. Dilute the solution to 100 ml. and polarize, observing the same temperature conditions as have been described in the preceding paragraph. The following table of constants must be used in connection with the Herzfeld formula. The readings should be reduced to terms of the normal weight of the material in 100 cc. of solution before making the calculations:

STEUERWALD'S TABLE OF CONSTANTS  
(Inversion at Room Temperature with 30 cc. Acid)

Invert- Reading in 200-mm. Tube	Temperature of the Observation = <i>t</i>						
	32° C.	30° C.	28° C.	26° C.	24° C.	22° C.	20° C.
—18	.....	.....	.....	.....	.....	145.51	145.54
—17	.....	.....	.....	145.39	145.42	145.46	145.49
—16	.....	145.27	145.30	145.34	145.37	145.41	145.44
—15	145.18	145.22	145.25	145.28	145.32	145.35	145.39
—14	145.12	145.16	145.19	145.23	145.26	145.30	145.34
—13	145.06	145.10	145.13	145.17	145.21	145.25	145.29
—12	145.00	145.04	145.08	145.12	145.16	145.20	145.24
—11	144.94	144.98	145.02	145.07	145.11	145.15	145.19
—10	144.88	144.93	144.97	145.01	145.06	145.10	145.14
— 9	144.82	144.87	144.91	144.96	145.00	145.05	145.10
— 8	144.77	144.81	144.86	144.90	144.95	145.00	145.05
— 7	144.71	144.75	144.79	144.84	144.90	144.95	145.00
— 6	144.65	144.70	144.74	144.79	144.84	144.89	144.95
— 5	144.59	144.64	144.69	144.74	144.79	144.84	144.90
— 4	144.53	144.58	144.63	144.68	144.74	144.79	144.85
— 3	144.47	144.52	144.58	144.63	144.69	144.74	144.80
— 2	144.41	144.46	144.52	144.58	144.63	144.69	144.75
— 1	144.35	144.41	144.46	144.52	144.58	144.64	144.70

<sup>2</sup> *Archief.*, 1913, 21, 831; *Int. Sugar Journ.*, 1913, 15, 489.

**226. Jackson-Gillis Clerget Modification.**—(Method IV.) A series of methods, using neutral salts in the direct polarization to offset the effect of the acid on the invert sugar in the invert polarization, were worked out by Jackson and Gillis of the Bureau of Standards.<sup>4</sup> Method IV is especially designed for use with cane products and has become widely used in cane-factory practice. The general method as given by the authors is quoted below, but details of the amounts of substance to be taken and the preparation of solutions for juices, sugars, molasses, etc., will be given in the chapters devoted to the analysis of these materials.

"Prepare a normal solution or a solution of such fractional normality as the nature of the substance and the sensibility of the saccharimeter requires. Make the solution to volume at the temperature at which the polariscopic observations are to be made. Clarify with the minimum quantity of dry basic lead acetate. Products of higher purity may be clarified by adding alumina cream before making to volume. Filter.

(If desired, the excess of lead may be removed at this point. Add pulverized potassium or sodium oxalate to complete precipitation of lead. The deleading reagent should be added to the whole filtrate. If the deleading is omitted, the lead is satisfactorily removed by the chlorides subsequently added.)

"Pipette two 50 ml. portions of the clear filtrate into two 100 ml. flasks and add 20 ml. of water to each flask. To one portion add 10 ml. of a solution of sodium chloride containing 231.5 grams per liter; make to volume at the temperature at which the observations are to be made and polarize in a water-jacketed tube. (Reading calculated to normal weight basis =  $P$ .) To the other portion add hydrochloric acid and invert by the method of Herbert S. Walker as follows: Insert a thermometer in a flask and heat by immersing in a hot-water bath until the temperature is exactly 65° C. Remove the flask from the bath, add 10 ml. of hydrochloric acid, 1.1029 sp. gr. at (20°/4° C.) = (24.85° Brix) mix by rotating and set aside for thirty minutes. Cool and make to volume at the temperature at which the observations are to be made. Polarize in a water-jacketed tube with temperature carefully regulated to the same as that for direct polarization. (Reading calculated to normal weight basis =  $P'$ ).

From Table 48 on page 524, find opposite the algebraic sum of  $P - P'$  (column No. 1), the value of the Clerget divisor (column No. 2). Apply the temperature correction. If the original solution was of fractional normality,  $P - P'$  must be multiplied by this fraction before selecting the value from the table. Divide the factor corrected for temperature into  $P - P'$  to obtain the Clerget sucrose.

**227. Clerget Method Using Invertase.**<sup>5</sup>—Commercial invertase preparations are available on the market.<sup>6</sup> If it is desired to prepare the solution in the laboratory, the procedure described under (1) may be used. The laboratory preparation may be further purified and concentrated by the ultra-filtration method described under (3).

(1) *Preparation of Crude Invertase Solution.*—Mix yeast with water in the proportion of 10 pounds of compressed baker's yeast with 5 liters of water. Add 2 liters of toluene and stir thoroughly at frequent intervals during the first twenty-four hours. Allow to stand for seven days with occasional stirring and filter by gravity through large fluted papers. Mix the residue with 2 liters of water, filter, and combine the filtrates. Purify<sup>7</sup> by adding

<sup>4</sup> Bureau of Standards Bulletin No. 375.

<sup>5</sup> Ass'n Off. Agri. Chem., Official Methods.

<sup>6</sup> Wallerstein Laboratories, 171 Madison Ave., N. Y. C.

<sup>7</sup> Jour. Ind. Eng. Chem., Vol. 16 (1924), 562.



15 grams of neutral lead acetate to each liter of extract and filtering on paper after all lead acetate has been dissolved. Complete the purification immediately by dialysis or by washing on the ultrafilter as directed under (3).

(2) *Preparation of a Collodion Ultrafilter.*<sup>2</sup>—Dissolve 6 grams of Cooper's negative cotton (snowy) in a mixture of 50 ml. of absolute alcohol and 50 ml. of absolute ether by first adding the alcohol to the cotton, allowing the mixture to stand in a stoppered flask for ten minutes, adding the ether, and shaking. Allow the solution to stand overnight, pour about 100 ml. into a 2000 ml. cylinder, and coat the entire inside surface of the cylinder with the collodion. Drain, and dry for ten minutes. Fill with water, let stand 10–15 minutes, pour out the water, and remove the collodion sack. Test for leaks by filling with water. Slit open longitudinally and cut out a circular piece about 7–8 inches in diameter. Cut the bottom from a 2-liter bottle or Erlenmeyer flask and grind the edge smooth. Place it upon the still moist collodion disc, fold the edge of the disk up around the bottle, and cement it thereto with collodion that contains an increased percentage of ether. Place three or four thicknesses of wet filter paper in an 8-inch Buchner funnel. Place the bottle with the collodion membrane upon the filter paper. Pour melted vaseline, to the depth of an inch, between the bottle and inside of the funnel. Provide the bottle with a small mechanical stirring device.

(3) *Washing and Concentration of Invertase Solution by Ultrafiltration.*—Filter 4 liters of the partially purified solution through the ultrafilter, stirring continuously, until about 1 liter remains. Wash with distilled water introduced by means of a constant level device until the filtrate is colorless, 3 or 4 liters of wash water being required. During the entire process the invertase solution must be preserved with toluene or chinisol.

(4) *Determining the Activity of the Invertase Solution.*—It is generally sufficient to test the activity of the invertase solution as follows: Dilute 1 ml. of the invertase preparation to 200 ml. Transfer 10 grams of sucrose (granulated sugar) to a sugar flask graduated at 100 ml. and 110 ml. dissolve in about 75 ml. of water, add 2 drops of glacial acetic acid, and dilute to the 100 ml. mark. To the 100 ml. of sugar solution add 10 ml. of the dilute invertase solution and mix thoroughly and rapidly, noting the exact time at which the solutions are mixed. At the termination of exactly sixty minutes make a portion of the solution just distinctly alkaline to litmus paper with anhydrous sodium carbonate and polarize in a 200 mm. tube at 20° C. If the invertase solution is sufficiently active, the alkaline solution will polarize approximately 31° Ventzke without correcting for the dilution of 110 ml. and the optical activity of the invertase solution.

**228. Determination with Invertase.**<sup>2</sup>—(a) *Direct Reading.* Dissolve the double normal weight (52 grams) of the substance in water in a 200 ml. flask; add basic lead acetate solution carefully, avoiding any excess, then 1–2 ml. alumina cream; shake; dilute to the mark with water; mix well; and filter, rejecting at least the first 25 ml. of the filtrate. Cover the funnel with a watch glass. When sufficient filtrate has collected, remove the lead from the solution by adding anhydrous sodium carbonate, a little at a time, avoiding any excess; mix well; and filter again, rejecting at least the first 25 ml. of the filtrate. (Instead of weighing 52 grams into a 200 ml. flask, two 26-gram portions may be diluted to 100 ml. each, and treated exactly as described. Depending on the color of the product, multiples or fractions of the normal weight may be used, and the results reduced by calculation to the basis of 26 grams in 100 ml.) Pipette one 50 ml. portion of the lead-free filtrate into a 100 ml. flask, dilute with water to the mark, mix well, and polarize in a 200-mm. tube. The result, multiplied by 2, is the direct reading (*P* of formula given below) or polarization before inversion. (If a 400-mm. tube is used, the reading equals *P*.)

<sup>2</sup> *Ibid.*, 170.

<sup>2</sup> Official Methods. Assn. Off. Agri. Chem.

(b) *Invert Reading*.—First determine the quantity of acetic acid necessary to render 50 ml. of the lead-free filtrate distinctly acid to methyl red indicator; then to another 50 ml. of the lead-free solution in a 100 ml. volumetric flask, add the requisite quantity of acid and 5 ml. of the invertase preparation, fill the flask with water nearly to 100 ml., and let stand overnight (preferably at a temperature not less than 20° C.). Cool, and dilute to 100 ml. at 20° C. Mix well and polarize at 20° C. in a 200 mm. tube. If the analyst is in doubt as to the completion of the hydrolysis, allow a portion of the solution to remain for several hours and again polarize. If there is no change from the previous reading, the inversion is complete, and the reading and temperature of the solution should be carefully noted. If it is necessary to work at a temperature other than 20° C., which is permissible within narrow limits, the volumes must be completed and both direct and invert readings must be made at the same temperature. Correct the invert reading for the optical activity of the invertase solution and multiply by 2. Calculate the percentage of sucrose by the following formula:

$$S = \frac{100(P - I)}{142 + 0.0676(m - 13) - T/2}$$

in which

$S$  = percentage of sucrose;

$P$  = direct reading, normal solution;

$I$  = invert reading, normal solution;

$T$  = temperature at which readings are made; and

$m$  = grams of total solids in 100 ml. of the invert solution read in the polariscope. (Solids by refractometer multiplied by sp. gr. of solution.) See p. 232.

## CHAPTER XIX

### CHEMICAL METHODS IN SUGAR ANALYSIS

#### "GLUCOSE DETERMINATION"

**229. Analytical Methods in General.**—The chemical methods of sugar analysis depend upon the property that dextrose, levulose, and other so-called reducing sugars have, in alkaline solutions, of reducing copper in the cupric state to cuprous oxide. Under like conditions, the amount of copper reduced is in proportion to the quantity of the reducing sugars present. In the table, page 531 the behavior of many of the carbohydrate bodies with alkaline copper reagent is given.

In the chemical methods of determining sugars (generally called glucose<sup>1</sup> determinations), it is very necessary to comply strictly with the directions for the analysis since a change in the conditions renders the tables for the calculations useless. In selecting the reduction method it must be taken into account that there is a slight reduction of copper by sucrose in the presence of invert sugar, so consideration must be given to the relative amounts of sucrose and reducing sugars in the material to be analyzed.

The successive steps involved after the selection of the method suitable for the material to be analyzed are: The preparation of the solution; the precipitation of the copper by boiling a measured portion of the glucose-containing solution with a fixed amount of Soxhlet's modification of Fehling's alkaline copper tartrate solution; a means of determining the amount of copper precipitated in the gravimetric methods, or of determining when the reduction is complete in the volumetric methods; and finally the calculation of the quantity of glucose present by means of tables or formulas suitable to the method employed. In general gravimetric methods are more accurate and the volumetric more rapid.

To determine sucrose by chemical methods it is first necessary to change it into invert sugar by hydrolysis with acids. If reducing sugars are already present these must be determined before the inversion of the sucrose and deducted from the total found after inversion, the sucrose being calculated from this difference. (See 252.)

It is the purpose of this chapter to give descriptions of various methods for the determination of glucose (reducing sugars) in general use, together with

<sup>1</sup> As is customary in the cane-sugar industry, this word is used throughout this book to include the dextrose and levulose in the material. The reducing-substances of cane and its products are various proportions of dextrose and levulose and possibly very small quantities of other reducing-substances. The mixed reducing-sugars are often termed "invert-sugar."

generalizations on the preparation of the solutions for the analysis. Specific directions as to which of these methods is to be employed with juices, sirups, molasses, sugars, etc., will be given in the special chapters devoted to the analysis of these materials.

**230. Preparing Solutions for Glucose Determinations.**—The preparation of the solution for the determination of glucose is of prime importance no matter what method is selected. This was demonstrated in some extensive studies made at the Central Control Laboratory <sup>2, 3</sup> (Cardenas, Cuba) of the Cuban-American Sugar Company at the request of Dr. Spencer. The use of normal lead acetate as a clarifying agent in glucose determinations had long been accepted as best practice because of the action of lead subacetate on levulose (Sec. 219) but the directions as to the quantity of lead to be used and the deleading agents to be employed allowed wide latitude. The investigations at Cardenas brought out the following points for work done on Cuban molasses and Cuban raw sugars:

1. The amount of normal lead acetate solution must be accurately measured and must be the minimum amount required to obtain a clear filtrate. Increasing the lead decreases the amount of glucose found.

2. Sodium sulphate and sodium carbonate should never be used as deleading agents as they give too low results.

3. Potassium oxalate and sodium phosphate in solution, and dry sodium oxalate, used as deleading agents give results checking each other though slightly higher than results using on lead whatever.

4. Where lead and deleading agents are used, if the lead precipitate is filtered off and the filtrate delead it will give lower results than if the deleading agent is added to the leaded solution and the combined precipitates are filtered out together.

5. Sodium phosphate and phosphoric acid are the only deleading agents that completely remove the lead. A small amount of lead left in solution does not disturb the precipitation of the copper; a large excess gives low results.

6. Clarification without lead, using kieselguhr only, gives consistent results and is a practical and simple method.

Clarification with kieselguhr without the use of lead has been employed for over ten years in the various laboratories of the Cuban-American Sugar Company with constant cross-checking showing excellent results. It must be remembered in using this method that the thin solution without lead or other preservative will deteriorate quickly, so determinations must be made immediately after the material is dissolved and filtered.

The only objection to this method is that there may be present copper-reducing non-sugars precipitable by lead, but in the work cited the indications were that no such substances were present. This was evidenced by the following results: Molasses treated with normal lead acetate and delead with sodium or potassium oxalate invariably gave results a little *higher* (1 to 2 mg. of copper precipitated) than are obtained with the no-lead and kieselguhr clarification; while the same molasses treated with the *oxalate only* and kiesel-

<sup>2</sup> Meade and Harris. Jour. Ind. Eng. Chem., Vol. 8 (1916), page 504.

<sup>3</sup> Harris. Jour. Ind. Eng. Chem., Vol. 13 (1921), page 925.

guhr gives the same high result as the lead and oxalate, showing that the disturbing influence is the oxalate and not the lead.

Eynon and Lane<sup>4</sup> proved that the presence of even small quantities of calcium salts is sufficient to cause a considerable decrease in the reducing power of glucose, particularly in the volumetric method bearing their name (Sec. 239). They recommend the removal of the calcium salts with sodium or potassium oxalate. Their findings show the reason for the higher results by Meade and Harris where oxalate was used. Since all cane products, particularly low-purity materials, contain some lime salts, it seems advisable where no lead is used to add some dry sodium oxalate with the kieselguhr before filtering. One-half a gram per gram of molasses is sufficient, or if preferred 5 ml. of 10 per cent potassium oxalate solution per gram of molasses may be added before making to the mark. A moderate excess of oxalate does not effect the copper-reducing powers. Eynon and Lane also found no indication of copper-reducing non-sugars precipitable by lead in the molasses and sugars they investigated, as evidenced by the fact that the use of lead for clarification and deleading with oxalates gave higher results than with no clarification at all (except kieselguhr). Oxalate alone checked their results with lead plus oxalate, proving that the lead had no effect on the reduction but that the oxalates removed the disturbing calcium salts.

In contradistinction to these findings and those brought out at Cardenas, Cook and McAllepe<sup>5</sup> working with Hawaiian molasses report that they "found decidedly lower results after treatment with lead acetate. That this was due to removal of non-sugar reducing substances was proved as follows: lead precipitate was absolutely freed from sugar by washing, decomposing by  $H_2S$ , and reprecipitating with neutral lead acetate after boiling out excess of  $H_2S$ , this process being repeated several times. The sugar-free precipitates were finally delead in several different ways, filtered, the filtrate added to Soxhlet solution, and heated under the conditions of the glucose determination. Copper was reduced in all cases. The amounts of these reducing non-sugars are sufficient to affect the results, hence lead clarification is necessary."

They corroborated the facts that lead is completely removed by disodium phosphate while lime is not, and that the oxalates remove all the lime but not all the lead. They recommend the use of a mixture of these salts in the proportion of 7 grams of disodium phosphate and 3 grams of potassium oxalate to 100 ml. using 1 ml. of this mixture for each gram of molasses. By this mixed solution all lead and all lime are completely removed.<sup>6</sup>

No matter what method of preparation of the solution is selected, it should be rigidly adhered to in all classes of determinations, otherwise comparable results cannot be obtained for use in calculating the "glucose balance" of the factory or refinery.

<sup>4</sup> Int. Sug. Jour. Vol. 25 (1923), page 305.

<sup>5</sup> Facts About Sugar. Vol. 13 (1928), page 298.

<sup>6</sup> An investigation now in progress by this writer (G. P. M.) indicates that the effect of normal lead acetate on reducing sugars is appreciable and that its use in any amount in preparing solutions for glucose determinations may introduce a considerable error.

**231. Copper Reagent.**—All the methods to be described here use the Soxhlet modification of Fehling's solution. This will be understood whether referred to as "Fehling's," "Soxhlet," or merely "the copper solution." It consists of two separate solutions which are mixed in equal volumes just before the analysis.

(a) *Copper Sulfate Solution.*—Dissolve 34.639 grams of copper sulfate ( $\text{Cu SO}_4 \cdot 5\text{H}_2\text{O}$ ) in water, dilute to 500 ml. and filter through prepared asbestos.

(b) *Alkaline Tartrate Solution.*—Dissolve 173 grams of Rochelle Salts (Sodium-potassium tartrate) and 50 grams of sodium hydroxide in water, allow to stand two days and filter through prepared asbestos.

#### GRAVIMETRIC METHODS FOR GLUCOSE (REDUCING SUGARS)

**232. Introductory.**—In the gravimetric methods the precipitated cuprous oxide is filtered out of the solution after the reduction (an excess of copper always remaining in the filtrate) and the copper is determined by any one of the methods to be enumerated later. From the amount of copper found the weight of reducing-sugars is determined from tables which have been calculated for the method employed. The amount of precipitated copper may be arrived at volumetrically but this does not make the method a "volumetric glucose" as generally understood. The volumetric methods depend upon determining the amount of the glucose-containing solution required to effect complete reduction in a fixed amount of Soxhlet solution.

**233. Herzfeld Method.**—(*Using Meissl-Hiller Factors.*) Extended experience has proved this to be an excellent method for all classes of cane-sugar products and its use is recommended for general routine work. The first step in the analysis after the preparation of the sample is the estimation of a suitable quantity of the material for the test. This need be done only in case of materials in which the approximate amount of glucose is not known, which is not generally the case in sugar-house routine work. Prepare a series of large test-tubes by adding successively 1, 2, 3, 4, and 5 ml. of the solution prepared as described above. Add 5 ml. of mixed Soxhlet's solution to the contents of each tube and heat to boiling during about two minutes. Allow the precipitates to settle. Compare the color of the supernatant liquid in each tube and note that which has the lightest tint, but is distinctly blue. Measure 20 times the volume of the dealed solution that this tube contained into a 100 ml. flask and dilute it to the mark with water. For example: Tube No. 3 is selected;  $3 \times 20 = 60$  ml. of the original solution to be made up to 100 ml. Use 50 ml. of this solution for the determination.

*Reduction to Cuprous Oxide.*—Measure 50 ml. of mixed Soxhlet solution (Sec. 231), i.e., 25 ml. of the copper solution and 25 ml. of the alkali solution into a 400 ml. Pyrex or other alkali-resistant beaker and add 50 ml. of the properly prepared sugar solution. Heat the contents of the beaker to the boiling point, taking approximately four minutes to reach this temperature, and continue the boiling with gentle ebullition for exactly two minutes. At the conclusion of the heating period add 100 ml. of cold recently boiled distilled

water, and immediately filter and collect the cuprous oxide, using one of the methods described further on.

The use of a thermometer to determine the beginning of the boiling period was recommended by Harris<sup>7</sup> and has been found to increase accuracy as well as to aid in the regulation of the pre-heating to the required four minutes. The thermometer is thrust through a rubber stopper which is held in the aperture of a perforated watch glass of suitable size to cover the 400 ml. beaker. The point of the thermometer should reach to within 5 mm. of the bottom of the beaker when the cover glass is in place. (See Fig. 83.) The moment when the thermometer registers 100° C. is taken as the beginning

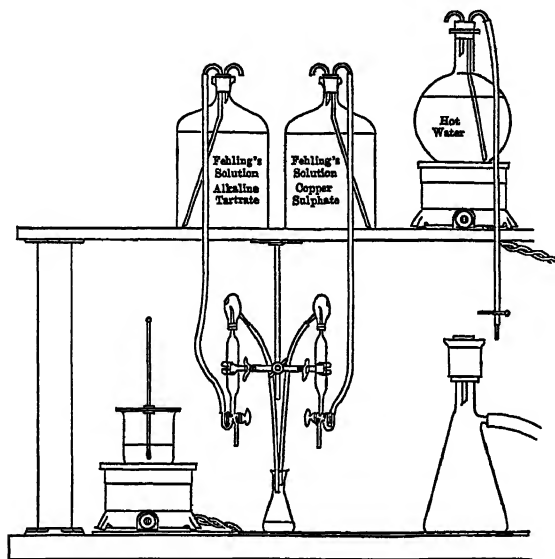


FIG. 83.—Arrangement for Glucose Determinations.

of the two-minute boiling period, although local ebullition sometimes starts before this temperature is reached while in other instances there is no evidence of boiling when the thermometer reaches 100°. An interval timer or accurate sand glass may well be used for timing the two-minute interval.

All the details of the method of preparing the solution and conducting the reduction must be strictly adhered to that the results may be comparable and approach the absolute glucose content. The beakers should be of Pyrex or similar glass, and all be of one size, preferably not larger than 400 ml. and of uniform thickness and diameter. The boiling should not be violent but only just apparent. The addition of cold water at the completion of the two-minute boiling period and the filtration should be prompt.

<sup>7</sup> Meade and Harris. Jour. Ind. Eng. Chem., Vol. 8 (1916), page 504.

*The Filtration of the Cuprous Oxide.*—The invention of the alundum filtering crucible and Spencer's method of making the joint at the rim of the crucible instead of the bottom as with the Gooch crucible, have greatly simplified this stage of the analysis. Other methods of filtration than with alundum are given for use in the absence of alundum ware.

(a) Provide a Spencer funnel (Fig. 84) or Sargent's alundum crucible-holder (Fig. 85). The latter is a modification of the Spencer funnel and may be used with any 60° funnel of suitable size. With the Spencer funnel the entire porous wall of the crucible may be thoroughly washed. The Sargent holder does not permit quite so thorough washing of the upper edge of the crucible, but, with care, the results are satisfactory. The funnel and holder must be of the proper size, otherwise the crucible itself will trap the washings and prevent prompt filtration. A brass funnel for use with the Sargent holder which permits very free filtration is shown in Fig. 86. A brass Spencer funnel may be readily made by one of the sugar-factory mechanics at a cost of a few cents, and is entirely satisfactory. The upper walls of the funnel must be parallel. The rubber rings are carried in stock by the dealers. These should be of pure soft rubber, and of about  $\frac{1}{4}$  inch

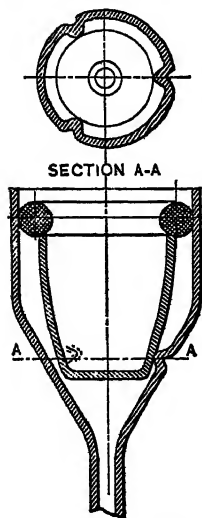


FIG. 84.—Spencer Crucible Holder.

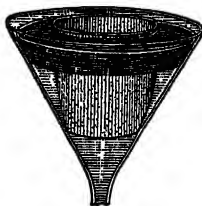


FIG. 85.—Sargent Crucible Holder.

cross-section. The funnel is placed in a suction-filtering heavy Erlenmeyer flask with side tubule. The suction is obtained by a filter-pump or preferably through a pipe communicating with the vapor-pipe of the multiple-effect evaporator. The alundum<sup>8</sup> crucibles require a very efficient filter-pump on account of their large filtering area.

The 25 ml. alundum crucible, porosity R A 360 has been found most satisfactory. New crucibles should be soaked in dilute nitric acid, thoroughly washed with hot water, then soaked in hot mixed Soxhlet's solution, and again thoroughly washed with hot water. Reverse washing is advisable both for new crucibles and for used crucibles between tests. The funnel

<sup>8</sup> Alundum crucibles may be substituted for platinum in most of the analytical work of the factory and agricultural laboratories. These lose weight very slowly in glucose work, due to the action of the alkali and the acid used for washing but remain practically at constant weight for the duration of the test.



shown in Fig. 86 is convenient for this purpose. Remove the Sargent holder and the spring brass cross in the bottom of the funnel and place the crucible upside-down on the rubber stopper. When suction is applied and hot water sprayed on the outside of the crucible the water is drawn through in the reverse direction from the filtration, freeing the pores of the alundum of all foreign matter. The crucibles can be freed of contained copper by soaking in hot dilute nitric followed by thorough washing. All crucibles after washing should be dried in an oven heated over a flame or preferably in an electric muffle and stored in a desiccator before use.

A Gooch crucible with asbestos mat may be used in place of alundum. The asbestos should first be digested for several days in dilute (1 : 3) hydrochloric acid washed free of acid, then digested for the same length of time with 10 per cent NaOH solution after which it is treated for several hours with hot alkaline tartrate solution. Then digest in nitric acid (1 : 3) wash free of acid with water and shake fiber up in water. About  $\frac{1}{4}$  inch asbestos mat is recommended in the crucible. The asbestos fiber improves with use. The copper adhering to the fiber is dissolved with strong nitric acid after which the fiber is thoroughly washed with hot water. A convenient form of laboratory layout for gravimetric glucose determinations is shown in Fig. 83.

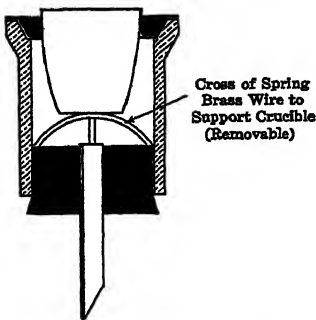


Fig. 86.—Brass Funnel for Use with Sargent Holder.

Immediately after the reduction to suboxide of copper, this precipitate is collected in the alundum crucible and is thoroughly washed with hot water. The crucible should be filled only about half full during filtration. The sub-oxide distributes itself on the walls of the crucible. The washing may be followed by moistening the oxide and crucible with pure alcohol to expedite the drying. Dry the crucible in an oven or cautiously over the flame of a lamp, according to the form in which the copper is ultimately to be weighed. Proceed by one of the following methods:

**234. Determination of the Copper.**—*Wedderburn's Method of Reduction to Metallic Copper.*<sup>9</sup>—This is the simplest of the methods involving reduction to metallic copper and its results are nearly as accurate as those by electrolysis and equal to reduction in hydrogen. Bend the wires of a pipe-stem or silica triangle to form a tripod support for the alundum crucible. Place the tripod in a metal beaker or other convenient metallic vessel. Cover the bottom of the beaker to a depth of about 1 cm. with alcohol. Denatured alcohol will serve. Place the beaker, covered with a watch-glass, on a hot plate and warm the alcohol until its vapors condense on the under side of the cover-glass. Heat the crucible to full redness, to burn off organic matter that may have been

<sup>9</sup> *Journal Ind. and Eng. Chem.* 7, 610. Original method, Vladimir Stanek, Z. Zuckerind. Boehmen, 32, 497; Votoček and Laxa, Abstract in Chem. Zeit. Chem. Repertorium, 21, 324.

carried down with the cuprous oxide; remove it from the flame and let it cool until the redness almost disappears; remove the cover from the beaker and place the crucible on the tripod and replace the cover. The oxide of copper is almost instantly reduced to metallic copper in the atmosphere of alcoholic vapor and adheres firmly to the walls of the crucible. The object of cooling the crucible to very faint redness is to prevent setting fire to the alcohol. If the alcohol should take fire the flames are readily extinguished by blowing upon them after covering the beaker. The beaker should be removed from the hot plate a moment after introducing the crucible. It is necessary to let the crucible cool for three or four minutes in the vapor of alcohol, after the reduction to avoid reoxidation of the copper. Should the crucible become quite cold, it should be moistened with pure alcohol and this be burned off. After cooling in a desiccator the crucible is weighed and the weight of copper is ascertained by difference. The whole operation consumes but five or six minutes and the copper plating is as good as that obtained by electrolysis.

Wedderburn's method may be conducted with a Gooch crucible, but the alundum ware is more convenient. An error may enter from occluded ash from the test solution or from carbon from the alcohol, but tests show this error to be very small and usually negligible. The calculation of the glucose is given farther on.

A special crucible furnace has been devised by Wedderburn for the preliminary heating. Any electric muffle will serve, or in the absence of current an alcohol-burner can be used.

*Electrolytic Method in Nitric Solution.*—Determining the copper electrolytically is never practiced in routine control tests. The practical accuracy of the various indirect methods has been demonstrated by numerous investigators so that electrolysis is resorted to only for checking methods and for research work.

Collect the cuprous oxide in an alundum crucible as described in Wedderburn's method, except that a glass funnel must be used and the crucible need not be tared. After washing the oxide very thoroughly, change the receiving-vessel for the filtrate. Let 4 ml. of concentrated nitric acid fall drop by drop upon the oxide, being careful that all parts of the latter are wetted by the acid. Follow the acid with a jet of hot water from a wash-bottle and wash the walls of the crucible thoroughly. Should any of the red oxide remain, repass the acid filtrate through the crucible. The oxide may conveniently be reduced to metallic copper as in Wedderburn's method and this be dissolved instead of the oxide. Transfer the filtrate to a 250 ml. beaker and dilute it to approximately 100 ml.

If a gooch crucible is used for the collection of the cuprous oxide, the asbestos mat and adhering oxide should be transferred from the crucible to the beaker by means of a glass rod. Dissolve any remaining oxide in the crucible with about 2 ml. of strong nitric acid from a pipette and rinse thoroughly with hot water allowing the acid and rinsings to flow into the beaker with the asbestos and dissolved copper. Heat the contents of the beaker until all copper is dissolved, filter through another prepared Gooch and wash thoroughly, diluting the filtrate to about 100 ml. as in the case of the alundum.

A convenient electrolytic apparatus may be made up in most laboratories with a large platinum dish (125–150 ml.), a heavy platinum wire bent in a flat spiral *S* as shown in Fig. 87 and a storage battery, either of the automobile or radio type in conjunction with a suitable rheostat. Connect the dish, *D*, containing the dissolved copper with the negative pole of the battery by placing it on a ring wrapped with the bare copper lead-wire. Insulate the ring from the support by means of a piece of rubber tubing *R*. When the spiral anode, *S*, is connected with the positive pole the copper will deposit on the inside of the dish. A current of 0.5 to 1.0 ampere is sufficient with stationary anode, the deposition of copper being hastened and made more even by having the solution heated to about 60° C. at the beginning of the electrolysis. Under these conditions the determination requires from one to three hours, depending on the amount of copper present. Rotation of the anode will greatly increase the rapidity of the operation, and permits of the use of 3 to 5 amperes current. If direct current from a lighting circuit is available it is the most convenient source for electrolytic work, using an ordinary rheostat or a bank of incandescent lamps to regulate the amount. Toward the end of the electrolysis, which becomes evident by the disappearance of the color, the solution should be tested for copper from time to time by withdrawing a drop and adding it to a little ammonia to neutralize the acid, then acetic acid to acidity and finally a drop of ferrocyanide of potassium solution, using a white porcelain plate to hold the solutions. When this solution no longer reacts for copper, *i.e.*, does not turn

brown when the ferrocyanide is added, without cutting off the electric current, withdraw the acid solution with a large pipette or by syphoning, at the same time replacing it with water. Repeat this operation until all the acid has been removed, then break the current, remove and dip the cylinder in pure alcohol and then in ether, and dry it in an oven for a few minutes. The current must not be discontinued so long as a trace of acid remains. The dish is now weighed, its increase of weight being due to the metallic copper deposited.

*Weighing the Copper as Cuprous Oxide.*—The red oxide collected on the alundum or Gooch crucible is completely washed with hot water as before and is then moistened with strong alcohol followed by a little ether, after which the crucible is heated in an oven at approximately 100°C. and dried thirty minutes, cooled and weighed. The weight of  $\text{Cu}_2\text{O} \times .888 =$  the weight of copper. This method has little to recommend it except simplicity. It should never be used with any but materials of high purity. Work with 96° raw sugars<sup>10</sup>

<sup>10</sup> Meade and Harris. *Loc. cit.*

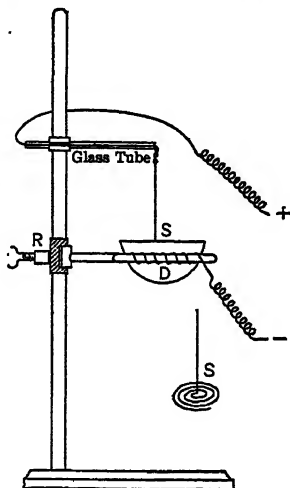


FIG. 87.—Apparatus for Electrolytic Copper Determinations.

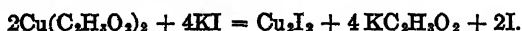
showed that results calculated from cuprous oxide were 10 per cent higher than those calculated from cupric oxide or metallic copper. The results obtained weighing as cuprous oxide with raw sugars were not much more accurate, based on percentage of the glucose content, than when working with final molasses.

*Weighing the Copper as Cupric Oxide.*—The crucible containing the red oxide is thoroughly washed with hot water as before. It is then dried and heated to full redness for fifteen minutes in a muffle furnace to oxidize the cuprous oxide to cupric. Cool the crucible in a desiccator and weigh as quickly as possible as the black oxide is very hygroscopic. The weight of  $\text{CuO} \times 0.8 =$  the weight of copper reduced.

This is an excellent method for routine work and long experience with it has proven its accuracy. It has been the practice in the laboratories which were under Dr. Spencer's direction, to determine the copper as cupric oxide first and then to reduce the oxide to metallic copper by the Vortoček-Wedderburn method and compare the two results, each method thus checking any possible error in the other. The metallic copper generally shows a fraction of a milligram higher, even in the most accurate work, due to occluded carbon from the alcohol.

*Low's Volumetric Thiosulphate Method.*—This is a highly accurate method for determining the quantity of copper precipitated which is used in investigations to check other methods. It is used in some industries for routine work and in the hands of a trained operator is rapid as well as accurate.

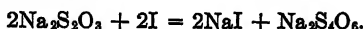
(1) Standardization of the Thiosulphate Solution.<sup>11</sup>—Prepare a solution of sodium thiosulphate containing about 19 grams of the pure crystals to the liter. Standardize as follows: Weigh accurately about 0.2 gram of pure copper foil and place in flask of about 250 ml. capacity. Dissolve by warming with 5 ml. of a mixture of equal volumes of strong nitric acid (sp. gr. 1.42) and water and then dilute to about 50 ml. Boil for a few minutes to expel partially the red fumes and then add 5 ml. of strong bromine water and boil until the bromine is thoroughly expelled. The bromine is to insure the complete destruction or removal of the red fumes. Remove from the heat and add a slight excess of strong ammonia water. Ordinarily it suffices to add 7 ml. of ammonia water of 0.90 sp. gr. Again boil until the excess of ammonia is expelled, as shown by a change of color of the liquid and a partial precipitation of the copper as hydroxide or oxide. Now add strong acetic acid in slight excess, perhaps 3 or 4 ml. of the 80 per cent acid in all and again boil for a moment if necessary to redissolve the copper precipitate. Cool to room temperature and add about 3 grams of potassium iodide or 6 ml. of a solution of the salt containing 50 grams in 100 ml. Cuprous iodide will be precipitated and iodine liberated according to the reaction,



The free iodine colors the mixture brown. Titrate at once with the thiosulphate solution until the brown tinge has become weak and then add sufficient starch liquor to produce a marked blue coloration. Continue the titration cautiously until the color due to free iodine has entirely vanished. The

<sup>11</sup> Bureau of Standards Circular No. 44, page 88.

blue color changes toward the end to a faint lilac. If at this point the thio-sulphate be added drop by drop and a little time be allowed for complete reaction after each addition, there is no difficulty in determining the end point within a single drop. One ml. of the thiosulphate solution will be found to correspond to about 0.005 gram of copper. The reaction between the thiosulphate and the iodine is:



The starch liquor may be made by boiling about 0.5 gram of starch with a little water and diluting with hot water to about 250 ml. The liquor should be homogeneous and free from lumps. It should be used cold and must be prepared frequently.

(2) Analysis. After washing the precipitated cuprous oxide, cover the Gooch crucible with a watch glass and dissolve the oxide by means of 5 ml. of warm nitric acid (1 : 1) poured under the watch glass with a pipette. Catch the filtrate in a flask of 250 ml. capacity and wash watch glass and Gooch crucible free of copper; 50 ml. of water will be sufficient. Boil to expel red fumes, add 5 ml. of bromine water, boil off the bromine, and proceed as in standardizing the thiosulphate.

*Calculation of the Percentage of Glucose for Herzfeld's Method by Meissl-Hiller Factors.*—If the material analyzed contains more than 1.5 per cent glucose and less than 98.5 sucrose in the contained solids calculate the percentage of glucose by the formula and factors given below. Tables have been expanded for products of known composition by E. W. Rice and others from these factors and formula, so that the tedious calculation is avoided for most routine work.

Let  $Cu$  = the weight of copper obtained;

$P$  = the polarization of the sample;

$W$  = the weight of the sample in the 50 ml. of the solution used for the determination;

$F$  = the factor obtained from the table for the conversion of copper to invert-sugar;

$$\frac{Cu}{2} = \text{approximate absolute weight of invert-sugar} = Z;$$

$$Z \times \frac{100}{W} = \text{approximate per cent of invert-sugar} = y;$$

$$\frac{100P}{P + y} = R, \text{ relative number for sucrose};$$

$$100 - R = I, \text{ relative number for invert-sugar};$$

$$\frac{CuF}{W} = \text{per cent of invert sugar}.$$

$Z$  facilitates reading the vertical columns; and the ratio of  $R$  to  $I$ , the horizontal columns of the table, for the purpose of finding the factor ( $F$ ) for the calculation of copper to invert-sugar.

**EXAMPLE.** The polarization of a sugar is 86.4 and 3.256 grams of it ( $W$ ) are equivalent to 0.290 gram of copper, Then:

$$\frac{Cu}{2} = \frac{.290}{2} = .145 = Z;$$

$$Z \times \frac{100}{W} = .145 \times \frac{100}{3.256} = 4.45 = y,$$

$$\frac{100P}{P + y} = \frac{8640}{86.4 + 4.45} = 95.1 = R;$$

$$100 - R = 100 - 95.1 = I = 4.9:$$

$$R : I = 95.1 : 4.9.$$

By consulting the table it will be seen that the vertical column headed 150 is nearest to  $Z$ , 145, and the horizontal column headed 95 : 5 is nearest to the ratio of  $R$  to  $I$ , 95.1 : 4.9. Where these columns meet we find the factor 51.2 which enters into the final calculations:

$$\frac{CuF}{W} = \frac{.290 \times 51.2}{3.256} = 4.56 \text{ per cent of invert-sugar.}$$

MEISSL AND HILLER'S <sup>11a</sup> FACTORS FOR THE DETERMINATION OF  
MORE THAN 1 PER CENT OF INVERT-SUGAR

Ratio of Sucrose to Invert- Sugar = R : I.	Approximate Absolute Weight of Invert-Sugar = Z						
	200 Mil- ligrams. Per Cent	175 Mil- ligrams. Per Cent	150 Mil- ligrams. Per Cent	125 Mil- ligrams. Per Cent	100 Mil- ligrams. Per Cent	75 Mil- ligrams. Per Cent	50 Mil- ligrams. Per Cent
0 : 10	56.4	55.4	54.5	53.8	53.2	53.0	53.0
10 : 90	56.3	55.3	54.4	53.8	53.2	52.9	52.9
20 : 80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30 : 70	56.1	55.1	54.2	53.7	53.2	52.6	52.6
40 : 60	55.9	55.0	54.1	53.6	53.1	52.5	52.4
50 : 50	55.7	54.9	54.0	53.5	53.1	52.3	52.2
60 : 40	55.6	54.7	53.8	53.2	52.8	52.1	51.9
70 : 30	55.5	54.5	53.5	52.9	52.5	51.9	51.6
80 : 20	55.4	54.3	53.3	52.7	52.2	51.7	51.3
90 : 10	54.6	53.6	53.1	52.6	52.1	51.6	51.2
91 : 9	54.1	53.6	52.6	52.1	51.6	51.2	50.7
92 : 8	53.6	53.1	52.1	51.6	51.2	50.7	50.3
93 : 7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94 : 6	53.1	52.6	51.6	50.7	50.3	49.8	48.9
95 : 5	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96 : 4	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97 : 3	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98 : 2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99 : 1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

<sup>11a</sup> Zeitschrift, 1889, p. 735.

**235. Herzfeld's Method for Materials of High Purity.** (*Containing less than 1.5 per cent glucose*).<sup>12</sup> Prepare a solution of the material so that it contains 20 grams in 100 ml. and clarify by filtering through kieselguhr. Using 50 ml. of this filtrate conduct the test exactly as given in Sec. 233 determining the amount of copper by any one of the methods given. From the copper found obtain the corresponding percentage of invert sugar from the table (p. 519.)

**236. Munson and Walker Unified Method.**—This is of wide application, the complete tables giving amounts of dextrose, invert sugar, invert sugar and sucrose, lactose, lactose and sucrose, and maltose corresponding to quantities of copper reduced when these various sugars are present. For our purposes invert sugar alone and invert sugar in the presence of sucrose are the only mixtures to be considered and the tables (p. 520) are abridged accordingly.

"If the composition of the mixture is known approximately, the weight of substance required can be judged without a previous assay. If less than 10 per cent of reducing sugar is present, a weight of 2 grams of total sugar should be taken in 50 ml. If more than 10 per cent, 0.4 gram in 50 ml. is the required amount. For unknown mixtures it is best to make an analysis with either one of the amounts of total sugar. This will serve either as a preliminary or final assay according to the amount present."<sup>13</sup> The analysis is conducted as in the two Herzfeld methods insofar as preparation of the solution, precipitation of the copper and collection and washing of the precipitate are concerned, except that no water is added at the end of the boiling period; also water at 60° C. is specified for washing the precipitate. This method is of great value for thin solutions of low purity such as press waters, char filter wash waters, and the like.

#### VOLUMETRIC METHODS FOR GLUCOSE (REDUCING SUGARS)

**237. Introductory.**—As opposed to the gravimetric procedure of determining the copper precipitated by a fixed amount of the glucose-containing solution, the volumetric methods determine the volume of test solutions required to precipitate completely all the copper in a certain amount of Fehling's solution. Volumetric methods are much used in routine tests because of their rapidity, but the gravimetric analyses are preferable for important control figures.

**238. Soxhlet's Volumetric Method.**<sup>14</sup>—"Make a preliminary titration to determine the approximate percentage of reducing sugar in the material under examination. Prepare a solution that contains approximately 1 per cent of reducing sugar. Place in a beaker 100 ml. of the reagent and approximately the quantity of the sugar solution necessary for its complete reduction. Boil for two minutes. Filter through a folded filter and test a portion of the

<sup>12</sup> Although generally described as being limited to materials of over 98.5 per cent sucrose, this is the accepted method for raw sugars and other materials of high purity, having a low percentage of glucose.

<sup>13</sup> Bureau of Standards Circular No. 44, pages 84 and 85.

<sup>14</sup> A. O. A. C. Official Methods (1925). Page 190

filtrate for copper by use of dilute acetic acid and dilute potassium ferrocyanide solution. Repeat, varying the volume of sugar solution, until two successive quantities are found that differ by 0.1 ml., one giving complete reduction and the other leaving a small quantity of copper in solution. The mean of these two readings is taken as the volume of the solution required for the complete precipitation of 100 ml. of the reagent.

Under these conditions 100 ml. of the reagent requires 0.494 gram of invert sugar for complete reduction. Calculate the percentage by the following formula:

$$\text{Percentage of invert sugar} = \frac{100 \times 0.494}{VW}, \text{ in which}$$

$V$  = the volume of the sugar solution required for the complete reduction of 100 ml. of the reagent; and

$W$  = the weight of the sample in 1 ml. of the sugar solution."

A table of reciprocals is given (p. 523) to facilitate the calculations.

The test for copper is best carried out by removing a small portion of the solution with a pipette, filtering through a small fluted filter-paper, allowing a drop to fall on a spot plate containing a mixture of 10 per cent acetic acid and potassium ferrocyanide (20 grams to a liter). A brown coloration indicates the presence of copper. The color test decreases in intensity as increased portions of the sugar solution are used. The progress of the test may be readily followed, after a little practice, by noting the appearance of the red oxide precipitate and the color of the supernatant liquid.

The volumetric method requires checking against a standard solution of invert sugar, the directions being as follows: Dissolve 4.75 of pure sucrose<sup>15</sup> in 75 ml. water, add 10 ml. of dilute hydrochloric acid (sp. gr. 1.1029 at 20° C./4° C. = 24.8° - 24.9° Brix at 20° C.) set aside for twenty-four hours at a temperature not below 20° C. (if temperature is above 25° C., ten hours will suffice). After the inversion is complete neutralize the acid with dilute sodium hydroxide solution mixing constantly while hydroxide is being added and then make up to 1 liter. Each milliliter of this solution contains 0.005 gram of invert sugar. Run a test with this pure invert solution in exactly the same manner as described above and calculate the strength of the copper solution in terms of invert sugar. Each new batch of Fehling's solution should be standardized in this way. The analyst thus standardizes his own procedure and subsequently rigidly adheres to that same procedure.

**239. Eynon-Lane Volumetric Method.**—(*Using Methylene Blue as Internal Indicator.*)<sup>16</sup>—This was published in 1923 and has rapidly found favor both in the sugar and glucose industries. It dispenses with the necessity of testing for the copper with ferrocyanide as in the Soxhlet method. The authors claim for it the accuracy of the gravimetric methods and the rapidity and simplicity are evident. The sugar solution is prepared of such a strength that it will contain from 0.25 to 0.8 gram glucose per 100 ml. if the sucrose is proportionately low (as in molasses) or from 0.1 to 0.3 gram if the sucrose is in

<sup>15</sup> A high-grade dry refined granulated sugar will do.

<sup>16</sup> Int. Sug. Journal. Vol. 25 (1923), p. 142.



greater proportion as in juices, sirups, sugars, etc. (See chapters relating to analysis of these products for specific instructions as to amount of sample.) Dry sodium oxalate is added to remove lime salts (see p. 239) and filtration is effected with kieselguhr; or normal lead acetate, deleading and deliming with mixed potassium oxalate and disodium phosphate as recommended by Cook and McAllep may be employed for clarification. After filtration the test is carried out as follows:

*Solutions.*—The Fehling's solution used is Soxhlet's modification and is prepared as in Sec. 231. The indicator is prepared by dissolving 1 gram of methylene blue in distilled water and making up to 100 ml. This solution will keep for months without change.

*Standard Method of Procedure.*—Ten or 25 ml. of mixed Fehling's solution is measured into an Erlenmeyer flask of 300 to 400 ml. capacity, and treated cold with almost the whole of the sugar solution required to effect reduction of all the copper, so that if possible no more than 1 ml. is required later to complete the titration. The approximate volume of sugar solution required is ascertained by a preliminary incremental titration which will be described further on. The flask containing the cold mixture is heated over a wire gauze; after the liquid has begun to boil it is kept in moderate ebullition for two minutes, and then, without removal of the flame, 3–5 drops of the methylene blue indicator are added, and the titration is completed in one minute further, so that the reaction liquid boils together for three minutes without interruption.

The indicator is so sensitive that the end-point can be determined to within one drop of the sugar solution in most cases. The complete decolorization of the methylene blue is usually sufficiently well indicated by the whole reaction liquid, in which the cuprous oxide is constantly churned up, becoming bright red or orange in color; but in case of doubt the flask may be removed from the wire gauze for a second or two and held against a sheet of white paper on the bench, when the edge of the liquid will appear bluish if the indicator is not completely decolorized. It is inadvisable to interrupt the boiling for more than a few seconds, as the indicator undergoes back-oxidation rather rapidly when the air is allowed free access to the flask, but there is no danger of this so long as there is a continuous stream of steam issuing from the mouth of the flask.

*Incremental Method of Titration.*—The method as outlined above is the one which permits the greatest precision and is least affected by personal factors. Since, however, the volume of sugar solution required must be known approximately in order that the whole of it may be added at one time before boiling, a preliminary titration is usually necessary. The method of carrying out this titration is as follows: 10 or 25 ml. of Fehling's solution, in a 300–400 ml. Erlenmeyer flask, is treated cold with 15 ml. of the sugar solution, and without further dilution is heated to boiling over a wire gauze. After the liquid has been boiling for about fifteen seconds, it will be possible to judge if the copper is all reduced, by the bright red color imparted the boiling liquid by the suspended cuprous oxide. If it is judged that nearly all the copper is reduced, a few drops of the methylene blue indicator are added, boiling is continued for

1-2 minutes, from the commencement of ebullition and then the sugar solution is added in small quantities, say 1 ml. or less at a time, the liquid being allowed to boil between successive additions for about ten seconds, until the color of the indicator is completely discharged. If after the mixture of Fehling's solution with 15 ml. of sugar solution has been boiling for about a quarter of a minute there still appears to be much unreduced copper, a further 10 ml. of sugar solution is added and the whole allowed to boil for a quarter of a minute, and so on until it is considered unsafe to add a further large increment of sugar solution; boiling is then continued for 1-2 minutes, after which the indicator is added and the titration is completed by small additions of the sugar solution. It is advisable not to add the indicator until the neighborhood of the end point has been reached, for the same reason that in the titration of iodine with thiosulphate solution the addition of the starch indicator is best postponed until as late a stage as possible, viz., because the indicator retains its full color until the end-point is reached and gives no warning to the operator to go slowly.

The flask should remain on the wire gauze over the Bunsen flame throughout the entire titration, except when it may be removed for a few seconds to ascertain if the end-point is reached. In adding the sugar solution to the reaction mixture the burette should be held in the hand and brought over the flask. The burette is fitted with a small outlet tube bent twice at right angles, so the body of the burette can be kept out of the steam while the jet is held over the flask. Burettes with glass stop-cocks are unsuitable for this work, as the cocks become heated by the steam and are very liable to jam.

#### DETERMINATION OF SUCROSE BY REDUCTION METHODS

First determine the glucose (reducing sugars) by the method of Munson and Walker (Sec. 236). Invert a second portion of the solution as follows: Pipette a 50-ml. portion into a 100-ml. flask and add 20 ml. of water, insert thermometer, and heat to exactly 65° by immersion in a hot water bath. Remove the flask from the bath, add 10 ml. of hydrochloric acid solution (24.85 Brix at 20° C.), mix and allow to stand for thirty minutes; then cool to room temperature in cold water, remove and rinse thermometer, and transfer the solution to a liter flask. Almost neutralize the acid with dilute caustic soda and complete the volume to 1000 ml. The solution should be so prepared that not more than 240 mg. of invert sugar is present in 50 ml. of the inverted solution. After the inversion is complete again determine the reducing sugars by the Munson and Walker method and deduct the amount obtained before inversion from that obtained after inversion. This represents the quantity of invert sugar obtained by the inversion of the sucrose, and since sucrose yields invert sugar on inversion at the rate of 100 : 95, this must be multiplied by 0.95 to calculate the sucrose originally.

(Per cent invert sugar after inversion — glucose in the material)  $\times$  0.95 = the percentage of sucrose.

## CHAPTER XX

### DENSITY AND TOTAL SOLIDS DETERMINATIONS

#### DENSIMETRIC METHODS

**240. General Remarks.**—The expression "density" is very commonly used in the sugar industry synonymously with "specific gravity." Sugar chemists also frequently term the degree Brix or the degree Baumé the "density" of the solution. This use of the word density is not strictly correct, but it is sanctioned by usage and the word will be used in this sense in this book for brevity and convenience. The graduations on hydrometers used in sugar-work are termed "degrees."

**241. Degree Brix or Balling.**—This system of hydrometer graduation was devised by Balling; the data were afterwards recalculated and checked by Brix. This hydrometer is known by both names in Germany, but the name "Brix" is used almost exclusively in America. The degree Brix is the percentage by weight of sucrose in a pure sugar solution. In commercial sugar analysis it is customary to consider the degree Brix as the percentage of solid matter, or the total solids, dissolved in a liquid. It is this feature of the Brix hydrometer, or spindle, as these instruments are commonly called, which renders it more convenient than the Baumé instrument in sugar-house work.

The degree Brix, as determined by floating a spindle in a sugar solution, is termed the apparent degree Brix. The percentage of total solid matter in a sugar solution as determined by drying in an oven is often called the true degree Brix. Except when qualified by the word "true," the apparent degree Brix is understood to be meant.

The French use two modifications of this instrument, the Brix-Dupont and the Brix-Vivien spindles. The Brix-Dupont hydrometer reads 0° in distilled water at 15° C., whereas the Brix spindle reads 0° at 17½° C., or according to the standard adopted by the International Congress of Chemistry, at 20° C. Both of these hydrometers indicate percentages by weight. The Brix-Vivien hydrometer indicates the percentage of sugar in a solution at 15° C. in terms of the weight and volume, *i.e.*, grams of sugar in 100 cc. of solution. The French modifications cited above are never used in the cane-sugar industry.

It may be well to state that the Brix spindle indicates percentages of sucrose in water solutions containing only the pure sugar.

**242. Degree Baumé.**—The Baumé (also spelled Beaumé) scale has no convenient relation with the percentage composition of any sugar-house product. The point to which the Baumé hydrometer sinks in distilled water at the standard temperature is marked zero; the corresponding point in pure sul-

phuric acid of 1.8427 specific gravity is marked 66°. Baumé spindles are also graduated for densities below zero, but the range of from 0° to 50° is all that is required in the sugar industry.

Gerlach and later Mateczek and Scheibler recalculated the values of the graduations of the Baumé scale. The recalculated numbers are termed the "new" or "corrected" degrees Baumé and are those given in the table, page 451.

The terms "new" and "corrected" are misleading since the most recent Baumé scale is that calculated by the Bureau of Standards<sup>1</sup> based on a constant multiplier or "modulus" of 145 according to the formula

$$\text{Degrees Baumé} = 145 - \frac{145}{\text{Specific Gravity}}$$

the specific gravities all being at 20° C. referred to water at 20° C. (or as commonly expressed sp. gr. 20°/20° C.). The Baumé degrees according to this scale are shown in the table on page 459. The so-called "new" or "corrected" (i.e., Gerlach) scale uses a modulus of 146.78 for specific gravities 17.5°/17.5° C.

The Baumé scale was at one time used almost exclusively in the sugar industry, but at present chemists prefer the Brix scale. Planters and sugar-makers still use the Baumé scale to some extent but not under conditions where great accuracy is demanded.

**243. Hydrometers or Spindles.**—These instruments are frequently termed "saccharometers" when specially graduated for use in the sugar industry.

A high-grade Brix hydrometer is shown in Fig. 88. This instrument is provided with a thermometer. Instruments for ordinary work, in the factory, are made of metal or of glass, and without the thermometer. In America and Germany the standard temperature for the graduation of these instruments has been, until recently, 17½° C. The present standard adopted by the International Congress of Applied Chemistry is 20° C. and this has very generally displaced the older standard. For temperatures varying from these standards, corrections must be applied to the readings.

Hydrometers whose normal or standard temperature is 17½° C., when floated in distilled water at this temperature, read 0°, and the corresponding specific gravity of the water is 1.0000. In other words, the weight of a volume of the sugar solution at 17½° C. is referred to the weight of the same volume of water at 17½° C. The table on page 451 is constructed for this normal temperature, 17½° C.

Hydrometers whose normal or standard temperature is 20° C., as specified by the International Congress of Applied Chemistry, when floated in water at this temperature, read 0°, and the corresponding specific gravity is 0.998234. The specific gravities of the solutions, corresponding with the degrees Brix, are given in the table, page 459.

It is to be hoped that the older standard of 17½° C. will soon become entirely obsolete in order that the duplication of tables, necessitated by the use of two standard temperatures, will be avoided.

<sup>1</sup> Bureau of Standards Circular No. 44, p. 159.

In using the hydrometer, it is floated in the sugar solution after this has been thoroughly mixed and allowed to stand until all air bubbles have risen

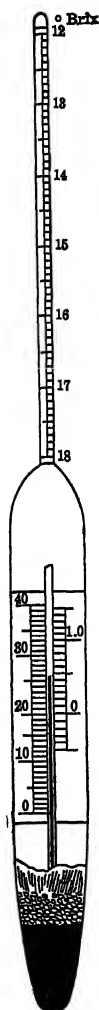


FIG. 88.—Brix Hydrometer.

to the surface. The instrument should be clean and dry and should be lowered carefully into the liquid so that the stem is wet for a short distance (2 or 3 mm.) above the point where it comes to rest. Suction may conveniently be used to assist in the removal of air, a glass tube connected with a vacuum line being thrust through a large rubber stopper, which is pressed down on the top of the cylinder for a few moments. The reading on the scale is made at the point *R*, not *R'*, of Fig. 89. The point *R* is at the level of the surface

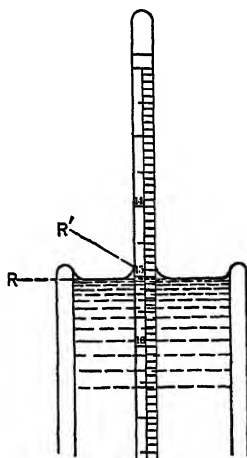


FIG. 89.—Reading the Hydrometer.

of the liquid and is selected for the reading, since the meniscus varies with the viscosity of the solution. It is often necessary in dark solutions to estimate the position of the point *R*. The reading of the scale is not made until after allowing sufficient time for the hydrometer to become of the same temperature as the solution. If the temperature of the liquid varies from the normal temperature for which the hydrometer is graduated, the observed reading of the scale must be corrected. For instruments whose normal temperature is  $17\frac{1}{2}^{\circ}\text{C}$ ., the corrections given in Gerlach's table, page 458, should be used. For instruments that are graduated at  $20^{\circ}\text{C}$ ., in conformity with the specification of the

International Commission for Uniform Methods, the table of corrections on pages 469 and 470 should be used. It is necessary in accurate work to use the hydrometer at near its normal temperature.

The type of Brix hydrometer shown in Fig. 88 has the thermometer

included and the corrections printed on the paper with the thermometric degrees, the height of the mercury column indicating the correction to be applied. This type of hydrometer has many points to recommend it. The temperature recorded is the same as that at which the Brix is taken; the use of reference tables is avoided and the standardization of the instrument includes both the hydrometer and the thermometer. On the other hand, such instruments are expensive and much more difficult to obtain of such accuracy that no correction need be applied. For these latter reasons some laboratories prefer a spindle of the same form but containing no thermometer, the temperature being taken with a floating thermometer immediately after the hydrometer is removed from the solution.

The stems of the hydrometers should be of small diameter in order that the graduation may be open and easily read. For accurate determinations the range of the instrument should not exceed 6° Brix marked in tenths, and the stem length should be at least 5 inches, such graduations permitting the reading of the Brix to 0.05° by dividing the smallest graduation with the eye. All hydrometers should be tested before being put into use against solutions of pure sucrose. For general laboratory purposes a high-grade granulated sugar may be used and the percentage of sugar determined with the polariscope. Hydrometers should be marked by the makers with a serial number for identification and to record the corrections found by the standardization.

Hydrometer jars or cylinders should be wide so that the spindle may float freely. A cylinder 2 inches diameter, 15 inches high, either with or without lip, is satisfactory.

244. *The Westphal Balance.*<sup>2</sup>—The principle of this balance, Fig. 90, may be briefly stated as follows: A glass bob is so adjusted as to be capable of displacing a given number of grams, five for instance, of distilled water at a given temperature when wholly immersed in the liquid and suspended by a fine platinum wire. The bobs may be graduated for any temperature; but for sugar-work 17½° C. or 20° C. (preferably the latter) are most convenient, since these are the temperatures usually employed in preparing specific-gravity tables. For accurate work the temperature of the solution whose specific gravity is to be determined should be exactly that for which the bob was graduated. The balance is provided with several riders or weights. Two of these riders, 1 and 2, are each exactly the weight of the water displaced by the bob at the standard temperature, 17½° C. The other riders, 3, 4, and 5, are respectively one-tenth, one-hundredth, and one-thousandth the weight of the first mentioned. When the weight 1 is hung on the hook at the end of the beam and the bob immersed in distilled water at 20° C. (or 17½° if the bob is so graduated), the balance should be in equilibrium, the weight having the value 1.000 in this position. In case the balance is not in equilibrium under these conditions, provided the bob has been correctly graduated, suspend this latter from the hook and turn the adjusting-screw until the pointers are exactly opposite one another. The weights 2, 3, 4, and 5 are placed on the beam in addition to 1 for liquids heavier than water, and have the values .1, .01, .001, and .0001, respectively, when placed on the corresponding graduations of the

<sup>2</sup> Adapted from Bulletin 13, Chem. Div., U. S. Dept. Agri.; also illustration.

beam, and for other graduations .300, .030, .003, .0003, etc. Each rider is provided with a hook from which additional weights may be suspended in case of more than one falling upon the same graduation.

The method of using the balance is as follows: Suspend the bob of the balance as described above in the solution, at the standard temperature, and weight the beam with the riders until the balance is in equilibrium. Read off

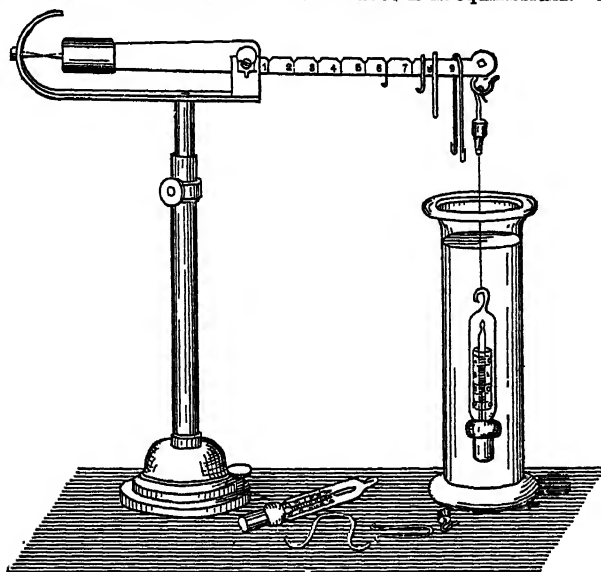


FIG. 90.—Westphal Balance.

the specific gravity from the position of the weights on the beam. Example: In determining the specific gravity of a sample of cane juice the position of the riders was as follows:

1 at point of suspension of the bob	= 1.000
2 not on the beam	
3 at 7.....	= 0.07
4 at 9.....	= 0.009
Specific gravity.....	= 1.079

The degree Brix or Baumé corresponding to the specific gravity may be ascertained from the tables on page 451.

**245. Pyknometers.**—Pyknometers are bottles so constructed that they may be filled with a definite volume of a liquid. Given the weight of this volume, it may be compared with the weight of an equal volume of distilled water. It is not often necessary to use a pyknometer in technical work, the more rapid density determinations by the hydrometer being usually sufficiently accurate.

Pyknometers are made in a great variety of forms. One of the most convenient of these is shown in Fig. 91. The side tube provides an outlet for the excess of liquid when the stopper, a fine thermometer, is put in place, also for the overflow, as the temperature of the liquid rises. The bottle should be filled with the liquid cooled to a temperature lower than that at which the density is to be determined. As the temperature rises to the desired point, the liquid expands and the excess is blotted with paper at the side tube. At the required temperature the cap is placed in position and receives any further liquid that may be expelled, as the temperature rises to that of the work-room. There is a minute opening in the top of the cap for the escape of the air.

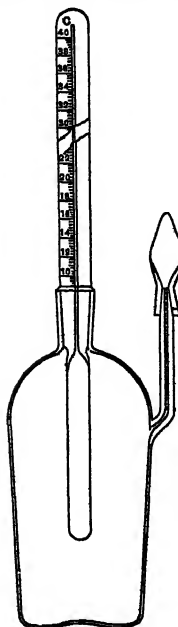


Fig. 91.—Pyknometer.

It is convenient in sugar-work to determine the specific gravity at  $17\frac{1}{2}^{\circ}\text{C.}$ , the solution at this temperature being compared with the weight of an equal volume of water at  $17\frac{1}{2}^{\circ}\text{C.}$  The International Committee on Uniform Methods of Analysis adopted  $20^{\circ}\text{C.}$  as the standard for the solution, referring it to water at  $4^{\circ}\text{C.}$ , the temperature of its maximum density. The standard used is indicated as follows:

$\frac{17\frac{1}{2}^{\circ}\text{C.}}{17\frac{1}{2}^{\circ}\text{C.}} = 1.07956$ , meaning that the temperature of the solution and that of the water were each  $17\frac{1}{2}^{\circ}\text{C.}$  The number above the line is that of the solution, and that below the line, that of the water.

By means of the pyknometer, described in this article, the weighings can readily be made of the liquid held by the specific gravity bottle at  $17\frac{1}{2}^{\circ}\text{C.}$  The specific gravity under these conditions is calculated by dividing the weight of the solution by the weight of an equal volume of water. In both cases the bottle is filled at  $17\frac{1}{2}^{\circ}\text{C.}$

In determining the specific gravity at the standard  $\frac{20^{\circ}}{4^{\circ}}\text{C.}$ , the process is more complicated, since the expansion of the glass pyknometer and the air density must be taken into account. The following description of the calculations, also the table, are from Landolt's work.<sup>3</sup> The pyknometer is first filled with recently boiled and cooled distilled water, and is weighed. The temperature and weight are noted. The weight is the apparent mass of the water in the air, a constant for the pyknometer at the temperature,  $t_0$ , at which it was filled and should therefore be determined with great care.

The calculations are made by the following formula, in which the letters have the values indicated below:

$$d_t = \frac{FQ_0}{W_0} + \frac{FQ_0}{W_0} \cdot 3\beta(t_0 - t) - \frac{\lambda(F - W_0)}{W_0},$$

<sup>3</sup>Optischem Drehungsvermögen.



$W_0$  = the apparent mass of the water in the air at the temperature  $t_0$ ;

$F$  = the apparent mass of the sugar solution in the air at the temperature  $t$ ;

$Q$  = specific gravity of water at the temperature  $t_0$ ;

$3\beta = 0.000024$ , the coefficient of cubical expansion of glass;

$\lambda = 0.0012$ , the air density;

$d_t$  = specific gravity of the solution of the sugar at the temperature  $t$  referred to water at  $4^\circ \text{C}$ .

The first factor of the formula is the uncorrected specific gravity, the second factor corrects the specific gravity for temperature and the third factor is for the reduction to weights in vacuo.

The value of  $Q$  may be taken from Landolt's table at the end of this article. If the difference between  $t$  and  $t_0$  is small, a mean value, 0.000024, may be used for  $3\beta$ .

#### SPECIFIC GRAVITY OF WATER AT VARIOUS TEMPERATURES

(From Landolt's Optischem Drehungsvermögen)

$t_0$	$Q_0$	$t_0$	$Q_0$	$t_0$	$Q_0$	$t_0$	$Q_0$
0	0.999874	17.7	0.998683	20.0	0.998235	22.4	0.997709
1	930	8	664	1	214	5	685
2	970	9	646	2	193	6	662
3	993	18.0	628	3	172	7	639
4	1.000000	1	609	4	151	8	615
5	0.999992	2	590	5	130	9	592
6	969	3	571	6	109	23	568
7	931	4	552	7	087	24	326
8	878	5	533	8	066	25	073
9	812	6	514	9	044	26	0.996811
10	731	7	495	21.0	023	27	540
11	637	8	476	1	001	28	260
12	530	9	456	2	0.997979	29	0.995971
13	410	19.0	437	3	957	30	674
14	277	1	417	4	935	31	368
15	132	2	397	5	913		
16	0.998976	3	377	6	890		
17.0	808	4	357	7	868		
1	790	5	337	8	846		
2	772	6	317	9	823		
3	755	7	296	22.0	800		
4	737	8	276	1	778		
5	719	9	255	2	755		
6	701			3	732		

## TOTAL SOLIDS BY DRYING

**246. General Observations.**—The determination of the total solids and moisture in sugar-house materials is one of the most unsatisfactory tests the chemist is called upon to make. This is due to the ready decomposition of several of the constituents and under some conditions to their tendency to occlude moisture. It is advisable to select methods that give fairly comparable results under average conditions and to use these at all times. If the drying is conducted in an air-oven, the same weight of material, the same size and kind of a dish, and the same temperature and heating-period should be adhered to at all times for a given class of materials. A temperature that is suitable, *e.g.*, to a high purity large crystal sugar is too high for a soft sugar of low polarization. The first requires a comparatively high temperature ( $105^{\circ}$  C.) to drive off the occluded water and the other a very low temperature to avoid decomposing the invert sugar.

Great care must be exercised in cooling and weighing the sample after drying as all dried sugar-products are very hygroscopic. It is best to employ covered dishes; to use a separate desiccator with each sample; to make sure that the desiccating material is active, and finally to weigh as rapidly as possible. Many control methods prescribe drying for a fixed time at a definite temperature, rather than to constant weight, since continued heating may cause a destruction of organic matter. Solids, such as sugars, are dried in the natural condition, while molasses, sirups and massecuites are generally accurately diluted 1 : 1 with water. This offers no extra manipulation since the "double dilution" of these products for Brix determinations is routine procedure. Juices and other thin solutions are weighed without dilution of course.

**247. Moisture Determinations in Ordinary Oven.**—*Sugars.*—Weigh 5 grams of the well-mixed sample into a flat dish (aluminum, 60 mm. in diameter with cover is convenient) spread the sugar evenly over the bottom and dry at  $105^{\circ}$  C. for three hours.<sup>4</sup> Cool in a desiccator and weigh. The loss in weight, divided by the weight of sugar taken, multiplied by 100 is recorded as percentage moisture.

*Liquid Products. Drying on Pumice-stone. Carr and Sanborn's Method.*<sup>5</sup>—Prepare pumice-stone in two sizes. One size should pass a 1 mm. sieve and the other should pass a 6 mm. sieve, circular perforations. Digest each with dilute sulfuric acid (1 : 9) for eight hours on a steam bath then wash free from acid and heat to dull redness. Place a layer of the finer pumice-stone 3 mm. thick on the bottom of a 60 mm. aluminum dish with cover then a layer of the coarse pumice-stone 6 to 10 mm. thick upon the first layer. Add such a quantity of the solution to the tared dish and pumice-stone as will

<sup>4</sup> The A. O. A. C. official method calls for drying ten hours at the temperature of boiling water; drying and weighing; then reheating for one hour periods until loss of weight is not more than 2 mgs. For large-grain sugars, heating for the last hour at  $105^{\circ}$ – $110^{\circ}$  is recommended. The shorter heating period given above is general practice in sugar laboratories.

<sup>5</sup> Bul. 46 Div. Chemistry U. S. Depart. Agri., p. 45.

yield approximately 1 gram of dry matter. In weighing the solution use a weighing-bottle provided with a cork through which a pipette passes. Dry to constant weight in a water-oven if the material contains little or no levulose, making trial weighings at intervals of two hours. The drying may be conducted in a vacuum-oven at about 70° C. in case of material containing much levulose or other readily oxidizable matter.

The weight of dry matter divided by the weight of solution used and the quotient multiplied by 100 = per cent of total solids.

*Drying on Quartz Sand.*—Place 25–30 grams of pure quartz sand that will pass a 40-mesh but not a 60-mesh sieve and a short stirring rod in a 60 mm. aluminum dish, dry thoroughly, cool in desiccator, and weigh. Then add sufficient of the diluted sample to yield approximately 1 gram of dry matter and mix thoroughly with the sand. Heat on a steam bath for 15–20 minutes, stirring at intervals of 2–3 minutes. Dry at 70° C. in a vacuum-oven, making trial weighings at two-hour intervals toward the end of the drying period until the change in weight does not exceed 2 mg. As in the method above the drying may be at atmospheric pressure in a water oven if the material contains little levulose or other decomposable substance.

In many sugar laboratories it is customary to determine moisture in molasses and other low-grade products at 100° C. without vacuum, in spite of the proportion of levulose present, using methods similar to the official methods given above. The heating time is generally fixed at five hours, no attempt being made to heat to constant weight.

Another drying method is to place the weighed solution directly in the dish with no pumice, sand or other inert material. A tendency to film over and occlude moisture makes such a procedure of doubtful value.

*A Convenient Vacuum-oven.*—Most laboratories are equipped with the ordinary electric drying ovens with thermostatic control, but electric vacuum-ovens are more costly. A steam-heated vacuum oven that can be made in the factory machine shop is shown in Fig. 92. Two pieces of large pipe (e.g., 8 inch and 12 inch) about 20 inches long make a convenient size. These should be welded or threaded to a collar at each end to form the steam jacket. One end is completely closed and the other has an air-tight removable door as shown. Connection is made with the factory's vacuum system by means of pressure tubing.

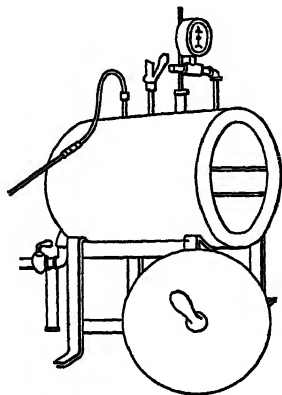


FIG. 92.—Vacuum Oven.

**248. Spencer Electric Oven for Moisture Determinations.**—The Spencer patent electric oven was first described in 1921.<sup>7</sup> It is made in two sizes and is a device for passing a large volume of air over a heating element and then

\* Adapted from Official Methods, A. O. A. C.

<sup>7</sup> Spencer: Jour. Ind. Eng. Chem., Vol. 13 (1921), page 70.

through a capsule containing the material to be dried. Type "A," used for sugars and sugar-containing liquids, is shown with the cover removed in Fig. 93 and also in section in Fig. 94. The larger size "B" will be described in the

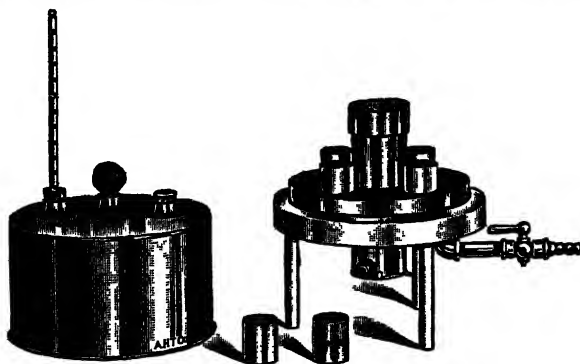


FIG. 93.—Spencer Drying Oven.

chapter on bagasse analysis. The aluminum capsules are 3 cm. in diameter by 4 cm. in depth and are fitted with a bottom of metal filter cloth to permit the air to pass freely. A study of the cross-section drawing will show the path of the air over the heating element and through the capsule.

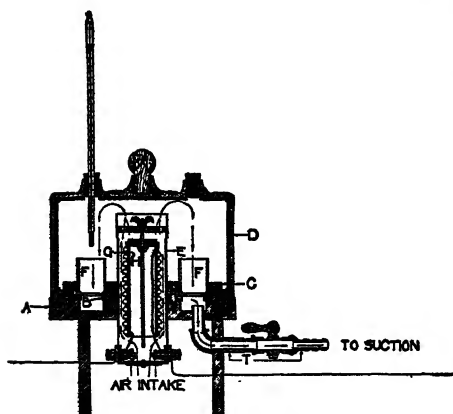


FIG. 94.—Cross-Section of Spencer Oven.

The heating element is connected in series with a sliding contact tube rheostat to regulate the temperature and also with a time switch to cut off the current at the end of the drying period. The current must never be turned on until after the suction is in operation nor must suction be stopped while the current is on, otherwise the heating element will burn out. To prevent this, Dr. Spencer devised a vacuum switch (Fig. 95) which cuts off the current when the suction is stopped.

*Moisture in Sugars.*—Four samples may be analyzed at once if desired but in case

less than four tests are to be made the unused capsule openings are blanked off with close-fitting corks. The capsules should be cleaned and dried and kept in a desiccator before use.

To make a test, weigh a dried capsule and then weigh into it as rapidly as possible 5 grams of the well-mixed sugar sample. Place the loaded capsule

in its opening in the oven, making sure that the seat is clean to avoid leakage of air around the capsule. Blank the unused openings and place the cover on the oven; open the suction (preferably a connection with a factory vacuum line) making sure that the cover fits tightly with no air leakage. The current is turned on and all resistance cut out so as to raise the temperature as quickly as possible to 110° C. As it approaches this heat slide the contact to a predetermined point and regulate the temperature by small adjustments of the rheostat. With practice, there is little difficulty in keeping the air current adjusted to within one or two degrees of 110°. The oven may be fitted with a thermostat if desired. Twenty minutes is the prescribed heating period, although the greater portion of the moisture is driven off in the first five minutes. After drying, cool in a desiccator and weigh.

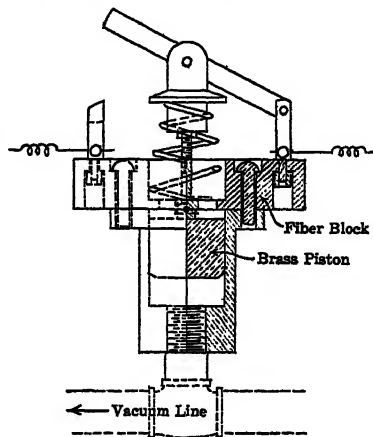


FIG. 95.—Vacuum Switch for Spencer Oven.

$$\text{Per cent Moisture} = \frac{\text{Loss in Weight}}{5} \times 100 = \text{Loss in Weight} \times 20.$$

*Moisture in Molasses, Sirups, Juices, etc.*—The Spencer oven may be used for the determination of moisture in liquid products by absorption of the material on asbestos.<sup>8</sup> Several precautions are necessary to insure concordant results. The asbestos should be freshly ignited and fluffy; the material should be diluted 1 : 1 (except in the case of juices or other thin solutions) so that it will be readily absorbed by the asbestos; not more than 4 ml. of liquid should be used, only one test should be made at a time, and the oven should be brought approximately to temperature (110°) before the loaded capsule is placed in it.

The procedure is as follows: The capsule is filled with fluffy freshly-ignited asbestos loosely packed and the whole heated in the oven at 110° for a few minutes, cooled and weighed. The capsule holds about 6 grams of asbestos. Using a weighing bottle fitted with a dropping pipette add about 4 ml. of the solution to be tested, a drop at a time, to the asbestos, being careful to add it slowly so that the asbestos will absorb it. Drop the liquid in the middle of the asbestos, not at the wall of the capsule, and allow the capsule to stand for a few minutes to complete the absorption. Examine the bottom of the capsule and if there is evidence of the liquid having gone through the asbestos discard the test and begin again. The oven is then heated to temperature, the capsule placed in position, suction again turned on and the air drawn through the sam-

<sup>8</sup> Meade: Jour. Ind. Eng. Chem., Vol. 13 (1921), p. 924.

ple for twenty minutes, temperature being maintained at 110° by rheostat or thermostat as in the case of sugars. One sample only should be tested at a time as it has been found that the air will not distribute itself evenly through two or more capsules, probably due to differences in the packing of the asbestos. After removing the capsule from the oven examine the bottom carefully, as the liquid sometimes pulls through in case the absorption by the asbestos has been faulty. To allow of the use of a larger volume of asbestos a special capsule with flaring sides has been made for testing liquids, but experience has shown that, with the precautions advised for adding the solution to the asbestos, the ordinary capsule will serve.

At the end of the heating period, cool and weigh the capsule as before. The calculation for materials diluted 1 : 1 is as follows:

Weight of capsule and material after drying minus weight of prepared capsule = solids in diluted solution.

Solids in diluted solution divided by weight diluted solution taken  $\times 100$  = per cent solids in 1 : 1 solution.

$100 - (\text{per cent solids in diluted solution} \times 2) = \text{per cent moisture in original material.}$

#### TOTAL SOLIDS BY REFRACTOMETER

**249. General Principles.**—Tolman and Smith's<sup>9</sup> investigations showed that most sugars in solutions of equal percentage composition have the same refractive indices, also that Gerlach's table of Brix hydrometer corrections may be used with small error to correct from room temperatures. Main<sup>10</sup> was the first to use the refractometer in refinery work. He found that the refractive index indicates accurately the content of solid matter in all but low products. Geerligs and van West<sup>11</sup> were the first to use the refractometer in cane-sugar factories. The table of Tolman and Smith, Main, and Geerligs, after temperature correction agree closely with one another. The refractometer is affected only by the solids in solution, therefore if the material under examination contains insoluble matter this is not included in the estimate of the solids. Within its limitations, *i.e.*, in materials containing only soluble solids and a small proportion of non-sugars, the refractometer is capable of giving very accurate indications as to the solids contents, usually quite as accurate as those by drying. The refractometric results with materials of low purity are usually intermediate between those by hydrometer and actual drying. In spite of certain advantages such as rapidity and the small amount of solution required for the test, the refractometer has not succeeded in displacing the older methods of hydrometers and solids by drying in general routine work. The cane-sugar maker, in order to adopt the refractometer in massecuite and molasses analysis, must completely change his idea of the suitable purities. These considerations and the conditions obtaining in Cuba deterred Dr. Spencer from applying

<sup>9</sup> J. Am. Chem. Soc., 1906, 28, 1476.

<sup>10</sup> Int. Sugar Journ., 1907, 9, 481.

<sup>11</sup> Archief, 1907, 15, 487. Abs. in Chem. Centralbl., 1908, 79 (1), 300.

the refractometer in the factories under his general superintendence of manufacture.

Three instruments are used in the refractometric estimation of the solids in sugar materials, viz.: (1) Abbé refractometer; (2) immersion refractometer; (3) sugar refractometer, a special form of Abbé instrument.

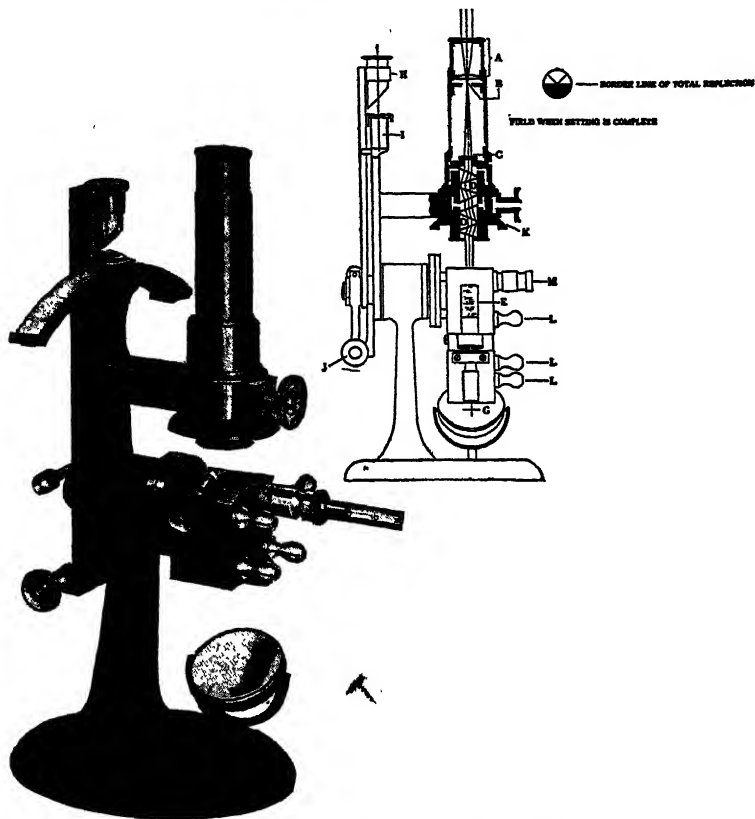


FIG. 96.—Abbé Refractometer (Bausch and Lomb).

(1) The Abbé Refractometer (as manufactured by Bausch and Lomb), Fig. 96, consists essentially of a polished flint-glass prism *E* and a ground flint-glass prism *F* of index  $N_D = 1.75$ , cemented into a metal mounting and a compensator consisting of two amici prisms *DD*. The mounting of one of the flint-glass prisms is hinged so that it may be separated from the other. A drop or two of the liquid to be tested is placed upon the polished surface of the fixed prism *E* and the hinged prism is carefully closed against it and the instrument is revolved to a convenient position with respect to the light. The

light, preferably from an electric lamp, is reflected upon the lower prism, *F*, by the mirror, *G*. The prisms are attached to a movable arm, at the left, which carries a scale-reader, *H*, for reading the index scale, *I*. To set the instrument, the movable arm is adjusted until the intersection of the reticule of the telescope cuts the line separating the dark from the light portion of the field the final adjustment being made by means of the screw *J*. The refractive index is then read off the scale with the scale-reader, *H*. Uniform temperature conditions should be maintained by means of a flow of water from a large vessel through the tubes, *LL*. The temperature of the observation should be noted and the correction be applied, using the table accompanying that of Geerligs, page 480. The percentage of dry matter corresponding to the index is given in Geerligs' table. The compensator, consisting of two small prisms *DD* placed in the telescopic tube, serves to eliminate the dispersion produced by the prisms. By rotating the small prisms with respect to each other by means of the milled head revolving the scale *K* the color is removed from the field. This compensation is necessary before making the observation. The prisms should be cleaned after use with filter-paper dipped in warm water and then be dried with soft linen. The instrument may be tested with air-free distilled water (index = 1.33298) or with standard plates.

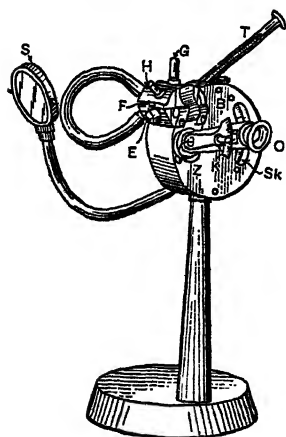


FIG. 97.—Zeiss Refractometer.

free distilled water (index = 1.33298) or with standard plates.

(2) The immersion refractometer is adapted to solutions containing up to 21.7 per cent of sugar and is not so well suited to general sugar-house work as the instruments (1) and (3) and therefore will not be described. Within its range it is more accurate than the other types and is quite suitable for juices of the usual range of density. Instructions and special tables are supplied by the dealers with the immersion instrument.

(3) *The sugar refractometer*<sup>1</sup> (made by Carl Zeiss of Jena), Fig. 97, is a special form of Abbé's instrument and has been designed especially for use in the sugar industry and the oil industry. It has the double prisms of the Abbé instrument and differs from this in the optical nature of the glass forming the prisms, designed by Schönrock, and in having a modified comparator.

*Construction.*—The new model consists of a circular casing supported on an upright and having rigidly attached to it the hinged double prism which receives the sample to be tested. The telescope is almost wholly enclosed within the casing, the ocular *O* only protruding outwards. It is carried by a stout radial arm. As in a polarimeter, the eye looks horizontally into the ocular, which to this end is mounted at a convenient height above the table. To save time in extensive control work, the double prism is so mounted that its inner surfaces are placed horizontally side by side when it is folded back for

<sup>1</sup> Abstracted from Zeiss' instructions.



cleaning and recharging. The hinge is at some distance from the body of the prisms, so as to protect it as far as possible from contact with the viscous juices. The image in the eyepiece presents the following appearance. In place of cross-lines, the field of view shows a circle with its center marked by a small black dot, on the right of it the percentage scale ranging from 0 to 50 per cent in fifths, thence to 97.5 per cent in tenths of a per cent, every integral of percentage being numbered, on the left of it a scale in terms of refractive indices (ranging from 1.330 to 1.540). The fact that the reading is taken in the field of view ensures a considerable saving of time. The instrument is equipped with a revolving compensator of the type first appended by C. Pulfrich to the dipping refractometer. This compensator is operated by the rotation of the knurled collar *K* and a completely colorless boundary line is obtainable with sugar juices of every possible concentration.

*Setting up the Instrument. Source of Light.*—Screw the cylindrical box upon the pillar and turn the eyepiece towards you. Then insert the arm of the mirror holder into its carrier at the back of the casing so that the mirror is on the left. For occasional measurements an adequate illumination is furnished by a bright sky, whilst direct sunlight renders it more difficult to take a reading. For rotational work affording comparable readings it is better to employ a 25 to 50 c.p. filament lamp with a frosted bulb. The latter should be suspended or, if mounted on a stand, placed behind the refractometer, that is, on the side away from the observer. Then give the mirror by trial a position in which it will throw a bright patch of light on the upper rectangular aperture *F* of the prism casing. It is only when taking readings by reflected light that the beam of light should be made to fall on the lower circular opening.

*Method of Applying the Sample.*—The first experiments should be made with light-colored solutions. The double prism should be opened and one or two large drops applied to the polished surface of the fixed prism with a well-rounded glass rod or fine tube, without, however, touching the latter as a precaution against accidental scratching. This being done the prism should be closed.

*Setting the Boundary Line.*—Rotate the collar *R* until the eyepiece is in its lowest position (0 per cent), follow the movement by a small displacement of the mirror until the field of view appears bright, and focus the eyepiece sharply upon the scale by screwing its mount in or out. The circle mark will then immediately appear sharply defined together with its center, which takes the place of the customary cross-lines. Move the eyepiece slowly upwards, following, if necessary, with the mirror (having only loosely clamped its arm) so as to maintain a bright field of view. A point will then be reached when the bright field appears traversed by an oblique line parallel to the scale lines, beyond which the field is more or less dark. Now proceed to render the boundary line colorless. To this end turn the collar *K* of the compensator until the boundary line appears colorless and apply a small correction to the eyepiece *O*, in order to set the boundary line accurately to the center of the circle mark. The boundary line should remain stationary when the adjustment is tested by inclining the mirror.

*Reading the Scale.*—The boundary line will now indicate on the right the

percentage of dry substance, and on the left the refractive indices. The latter scale, it will be seen, is ruled and figured in terms of the third decimal. For example, there are nine division lines between 1.41 and 1.42, *i.e.*, ten intervals or units of the fourth decimal. At the time of taking the refractometer reading the temperature indicated by the thermometer should likewise be noted.

*The Examination of Very Dark Juices.*—When examining a very dark sample, if the lighter half of the field should be so dark that it becomes impracticable to set the instrument accurately to an interval of the scale, recourse must be had to illumination by reflected light. To this end the mirror should be lowered until it causes the bright light to fall upon the round window *T* of the fixed prism. The intensity relation of the two halves of the field of view at once reverses. The upper half, which previously was the darker half, being illuminated by the rays which have been totally reflected by the sample, appears brighter than the lower half, which, being illuminated by the partially reflected rays, is still relatively bright. The degree of brightness changes suddenly from one-half to the other as soon as the compensator has been carefully set in its most favorable position. On the other hand, the boundary line upon which the reading is taken can be intentionally blotted out by a false position of the compensator. One not accustomed to the use of the instrument should study the appearance of the boundary line on a light-colored sample, which in the first instance he should view in transmitted light, as described in art. (c). In the case of very dark oils or juices (molasses) the boundary is most easily found by a movement of the eyepiece.

*Examination of Viscous Solutions.*—Slightly warm the prisms by circulating water through the mounting. Apply the solution to the prism and promptly make the observation.

## CHAPTER XXI

### DETERMINATION OF ASH

**250. Choice of Method.**—A marked change of ideas concerning ash determinations has occurred during the last ten or fifteen years. The "sulphated ash" was always used for routine work, largely on account of its simplicity, but the "carbonated" or "normal ash" (*i.e.*, direct incineration) was considered more accurate and the only method to be employed for accurate work or investigations. Work by Withrow and others showed the difference between the sulphated ash and the ash by direct incineration was much greater than the conventional 10 per cent usually deducted to reduce the sulphated ash to the normal ash. Withrow and Jamison<sup>1</sup> found that for the Cuban raw sugar used in their experiments 33.25 per cent was the correct deduction. Dr. Spencer had a series of determinations made by the two methods of the ash in final molasses from several Cuban factories and found that the sulphated ash ranged from 17 per cent to 31 per cent higher than the normal ash. These and many similar investigations led to the conclusion that the sulphated ash was too unreliable for use, and recommendations were made to the Assn. of Official Agricultural Chemists and in many of the journals that its use be discontinued.

A complete reversal of ideas occurred through the publication by Browne and Gamble<sup>2</sup> which proved that the direct incineration method is the unreliable one of the two, because of the loss of chlorine, sulphur and other constituents to a varying and uncontrollable degree. Lunden<sup>3</sup> also concluded that the sulphate method gave the more correct measure of the ash. It is now generally recognized that neither method gives the absolute measure of the salts present but that the sulphated ash is a relative measure of the salt content which is reproducible in check tests. For this reason all work with conductimetric ash has been with a view to checking the sulphated ash.

**251. Sulphated Ash Method.**—Weigh 2 to 5 grams of the material into a 50–100 ml. platinum dish (amounts of sample to be taken will be specified in chapters dealing with analysis of specific products, *i.e.*, juice, molasses, sugars, etc.), add 0.5 ml. of concentrated sulphuric acid, heat gently until the sample is well carbonized and then heat in a muffle at low red heat until all carbon is burned off. Cool and add a few drops more of sulphuric acid, heat until this is fully volatilized, cool and weigh. Withrow and Jamison<sup>4</sup> advocate the

<sup>1</sup> *Ind. and Eng. Chem.* Vol. 15 (1923), No. 4.

<sup>2</sup> *Facts about Sugar.* December 15, 1923.

<sup>3</sup> *Z. Ver. deut. Zuckerind.* Vol. 75 (1925), p. 774.

<sup>4</sup> *Loc. cit.*

use of sulphuric acid diluted 1: 1. They found porcelain crucibles suitable for this work but objected to fused silica as giving low results.

The A. O. A. C. specifies that the weight of ash found shall be recorded without any reduction as sulphated ash. The usual practice in sugar work is to deduct 10 per cent. Obviously either procedure may be adopted provided it is followed consistently for all products. The work on conductimetric ash is all calculated to check sulphated ash with the one-tenth deduction.

**252. Direct Incineration, "Normal" or "Carbonated" Ash.**—Weigh out the sample as above and heat slowly until swelling ceases. (The addition of a few drops of pure olive oil is advocated by the Assn. Official Agricultural Chemists.) Then heat in a muffle at low redness (Withrow says 500° C.) until a white ash is obtained; moisten with a little ammonium carbonate and again heat in muffle at low red heat. As stated above the method is used only in investigation work and is difficult if not impossible to check.

It is difficult to burn sugar or sugar-house products to obtain a large quantity of the carbonated ash for complete analysis. The usual method is conducted as follows: The material is heated in a large platinum dish until it takes fire and the flames burn out. It swells greatly and is difficult to confine to the dish. (Pure olive oil or vaseline may be added to reduce this danger.) After the material has been sufficiently charred, it is transferred to a glass mortar and is rubbed to a powder. The powder is washed upon an ashless paper filter and is thoroughly extracted with hot water. The filtrate is reserved for further treatment. The filter and insoluble matter are returned to the platinum dish and are completely ashed. The filtrate is now evaporated to dryness in the platinum dish and the dish and contents are then heated to low redness to burn off any remaining organic matter. The complete analysis of the ash so obtained is carried out according to the accepted methods of quantitative analysis.

#### ELECTROMETRIC ASH DETERMINATIONS <sup>4a</sup>

**253. Introductory.**—Some time after Arrhenius' fundamental investigations on the electrical conductivity of salt solutions containing non-electrolytes, the conductivity of sugar products and its relation to their ash content was studied in Europe by several investigators (about 1909-1910). Then the subject rested a number of years, and interest in it has only lately been revived by the work of Snell <sup>5</sup> on maple products, by Lunden <sup>6</sup> and by Toedt <sup>7</sup> on beet products, and by Zerban and Mull <sup>8</sup> and Zerban and Sattler <sup>9</sup> on cane products.

The advantages of electrometric ash over the chemical method lie chiefly in its speed and accuracy and simplicity.

<sup>4a</sup> Abstracted from descriptions supplied by Drs. Zerban and Sattler.

<sup>5</sup> Jour. Assoc. Official Agr. Chemists, **4**, 435 (1921).

<sup>6</sup> Cent. Zuckerind. **33**, 204 (1925); Z. Ver. Deut. Zuckerind., **75**, 763 (1925).

<sup>7</sup> Z. Ver. Deut. Zuckerind., **75**, 429 (1925).

<sup>8</sup> Facts About Sugar. March 20, 1926.

<sup>9</sup> Facts About Sugar, Dec. 4, 1926; Oct. 8, 1927; July 21-28, 1928.

The results as determined with high-class apparatus are comparable in accuracy with sulphated ash determinations made under most exacting research conditions, and may be made in about one-tenth the time. Conductivity measurements are particularly useful in refinery investigations of ash adsorption by boneblack.

The analysis of sugar for ash by the electrometric method is based on the fact that distilled water is an exceedingly poor conductor of electricity, as is a highly purified solution of sucrose. Soluble mineral impurities, as well as organic salts, which are found in raw sugar, break up in water solution into electrically charged particles called "ions" and as the ease with which electricity passes through a solution depends upon the relative amounts of the ions present, it is possible to estimate the percentage of mineral matter or "ash" in a sample of raw sugar by determining the conductivity of its solution. Resistance is the inverse of the conductivity, and it is the resistance of the test solution that is actually measured.

**254. Method.**—*Determination of Cell Constant.*—The first step necessary in the operation of the sugar ash bridge is the determination of the cell constant for the cell in question. This factor changes from cell to cell, as well as from time to time in the same cell. Whenever the cell has been changed or cleaned or in any way tampered with, the cell constant must be redetermined.

Using a bridge specially designed for sugar work, no calculations of the cell constant is necessary, as this is done in effect by the bridge itself.<sup>10</sup> (The apparatus shown in Fig. 98 is the Leeds and Northrup "Sugar Ash Bridge.") In the determination of the cell constant, the resistance dial is set to the theoretical calculated resistance of the standard KCl solution, (either N/50 or N/100), the temperature compensating dial is turned to the temperature corresponding to the thermometer reading, the current is switched on (alternating current is essential) and the cell constant dial is adjusted until the system is in equilibrium as shown by the galvanometer. The value thus found is the cell constant, and the dial is locked at this setting so that the instrument reads in true reciprocal ohms at any subsequent setting.

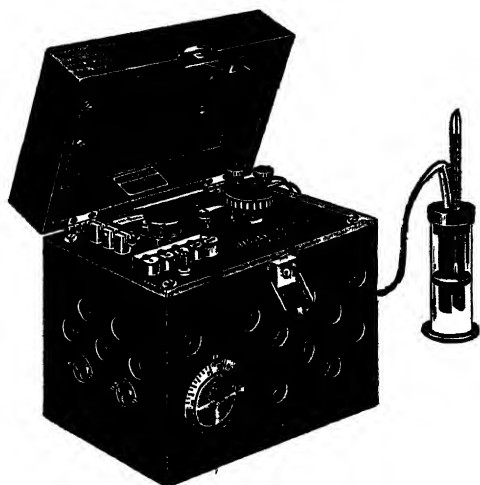


FIG. 98.—Sugar Ash Bridge.

<sup>10</sup> For directions concerning the assembling of bridges and cells, consult original articles by Zerban and his associates.

*Determination of Conductivity of Solution.*—Twenty-five grams of a well-mixed sample of the sugar is washed into a 500 ml. flask with conductivity water, dissolved, and then poured into the cell up to the "40" mark after several rinsings with the solution. The cell constant dial being locked in the proper position, the temperature adjusting dial is turned to the temperature indicated, and the resistance dial is then rotated until the galvanometer shows zero deflection. The instrument now reads specific conductivity of the solution.

*Calculation of the Ash.*—The percentage of sulphated ash is calculated by means of the equation, per cent ash =  $C \times K$ , where  $K$  is the conductivity, and  $C$  a constant. This constant is widely variable according to districts, as well as to different centrals in the same district, and must be determined by each factory for its own sugar. The best way to determine ash is to construct a curve, made by the chemist from a number of representative samples of the sugar, plotting conductivity (as experimentally determined) against sulphated ash, minus one-tenth (also as experimentally determined). The value of  $C$  for the above equation for different localities is shown in the following table:<sup>11</sup>

Camaguey, Cuba.....	1743	British West Indies.....	1560
Habana, Cuba.....	1730	Santo Domingo.....	1802
Matanzas, Cuba.....	1755	Philippines.....	1816
Oriente, Cuba.....	1796	Porto Rico.....	1672
Pinar del Rio, Cuba.....	1689	Hawaiian Islands.....	1630
Santa Clara, Cuba.....	1765		
All Cuba.....	1757		

As can be seen from the above table, the factor differs widely for different grades of sugar, and also differs widely for different factories in the same locality, but if the factor for any particular factory is determined as suggested above, it will be found to be entirely satisfactory, giving consistently correct results. In the case of refineries continuously melting certain marks, or in the case of a central laboratory analyzing sugars from several factories, a curve would be made, or the factor determined, for each mark.

*Acid Conductivity Method.*—The discrepancies in the value of the constant as shown above is due, among other things, to the variation in the composition of dissolved salts. This has been studied by Sattler and Zerban<sup>12</sup> and they have worked out a method whereby this factor is eliminated, and the ash can be calculated from the equation

$$\text{Per cent ash} = 0.0001757 \times (9.13 K + 1935 - K_1),$$

where  $K$  is the conductivity of the sugar solution, determined as described above, and  $K_1$  is the conductivity of the sugar in acid solution. When the ash is determined by this method, the usual discrepancies attendant to the first method are not found, and the equation can be used for all classes

<sup>11</sup> Zerban and Sattler. *Facts About Sugar*. Dec. 4, 1926.

<sup>12</sup> *Loc. cit.*

of sugar with negligible variations between the calculated results and the experimental chemical ash.

In this method, the solution is prepared in the same way as in the single-conductivity method, and the first (water) conductivity is made in the same way. Then a 200 ml. portion of the solution (25/500) is introduced into a Kohlrausch flask, 5 ml. of N/4 HCl, accurately measured, are added, and the flask shaken to thorough mixing of the contents. This solution is then placed in the cell, and the conductivity is again determined. These two conductivities are inserted in the equation, and calculated to per cent sulphated ash. In setting the temperature compensating dial on the L. & N. Sugar Ash Bridge for readings "with acid," the settings must be slightly changed as follows:

Temperature of solution.....	20°	22.0°	24.0°	26.0°	28.0°	30.0°
Correct dial setting.....	20°	21.5°	23.1°	24.6°	26.2°	27.7°

The hydrochloric acid (N/4) used must be very carefully adjusted as to concentration. Titration against standard alkali by the use of indicators is not accurate enough for this purpose. It is best to control the strength of the acid by its specific conductance. A mixture of 200 ml. of conductivity water and 5 ml. of the acid used in this work should have a corrected specific conductance of 0.002370 at 20° C. Care must be taken to cleanse the electrodes thoroughly after measuring the solutions containing the acid. This is best accomplished by washing once with dilute pure methyl alcohol, about 20 per cent by volume, and then rinsing with water.

**255. Conductivity Water.**—The first distillation should be made over alkaline potassium permanganate in any convenient still after which the water is redistilled in a Pyrex flask using a condenser of block tin. Usually, after the first still has been in use for some time, the water issuing therefrom will be found to have a specific conductance equal to the redistilled water, namely, of about  $1.2$  to  $1.4 \times 10^{-6}$ . When this state exists, the redistilling may be discontinued, as water of lower conductivity deteriorates rapidly, and besides, this conductivity is quite good enough for this type of work.

## CHAPTER XXII

### HYDROGEN-ION CONTROL

**256. Advantages of H-ion Control.**—The colorimetric determination of hydrogen-ion concentration (pH) as a measure of the reactions of juices, sirups and other sugar solutions has become an essential part of the control in most cane sugar factories and refineries. It has many advantages over the old system of test papers and tirations; most important of which is that hydrogen-ion concentration is the measure of the *intensity* of the acidity or alkalinity whereas the older methods measured only the *quantity* of acidity or alkalinity. Since the sucrose inverting power of an acid is a direct function of its degree of dissociation (*i.e.*, of the amount of hydrogen-ions in solution) the importance of this advantage in the sugar industry cannot be too greatly stressed. In fact, this relationship is so exact that the rate of inversion of sucrose has been used as a measure of the H-ion concentration of bio-chemical solutions.<sup>1</sup> Another advantage of the pH control is the simplicity of the colorimetric determination. Unskilled workmen can readily be trained to make the test, and the numerical expression, or pH number, is easily understood by them. The fact that the test is as accurately carried out by artificial light as by daylight is an added advantage over test papers and titrations.

**257. Theory of Hydrogen-ion Concentration.**—No theoretical knowledge of the subject is necessary to the use of colorimetric methods but a concise statement of the principles involved is here given. In all aqueous solutions it has been proved by electrometric measurements that the product of the hydrogen-ion concentration (H+) and the hydroxyl-ion concentration (OH-) always equals a constant.

$$(H+)(OH-) = \frac{1}{100,000,000,000,000} = 10^{-14} \dots (1)$$

Therefore, no matter what the concentration of hydroxyl-ions in the solution, there must be sufficient hydrogen ions to satisfy the equation, so that we are able to speak of the hydrogen-ion concentration of alkaline solutions, since by so doing we are also stating indirectly the hydroxyl-ion concentration.

The direct expression of hydrogen-ion concentration gives a series of unwieldy fractions and to avoid these Sorensen in 1909 suggested the use of the logarithm of the reciprocal of the H-ion concentration, which he found to be a

<sup>1</sup> "The Determination of Hydrogen-ions." Clark, p. 296.



function of the potential of the hydrogen electrode in determining hydrogen-ion concentration electrically. The equation

$$\text{pH} = \log \frac{1}{(\text{H}^+)} \quad . . . . . (2)$$

where  $(\text{H}^+)$  represents the concentration of H-ions in gram-atoms per liter, defines H-ion concentration quite as well as  $(\text{H}^+)$  itself. As pH is the logarithm of the reciprocal of a power of 10, it follows that it equals numerically the exponent or power to which 10 is raised. For example, in absolutely pure distilled water the H-ion concentration and the OH-ion concentration are exactly equal and since according to equation (1) the product of the two must be  $10^{-14}$  the concentration of each must be  $10^{-7}$ . Hence by equation (2)

$\text{pH} = \log \frac{1}{10^{-7}}$  and since  $\log \frac{1}{10^{-7}} = 7$ ,  $\text{pH} = 7.0$  which represents neutrality on the pH scale.

If the H-ion concentration exceeds the OH-ion concentration, the solution is acid and the pH number is below 7.0, while if the reverse is true—i.e., if there are more hydroxyl-ions than hydrogen-ions in the solution—the solution is alkaline and the pH is above 7.0. Thus it is seen that the pH numbers form an acidity-alkalinity scale in which the *lower* the pH the *higher* the hydrogen-ion concentration (the pH numbers being reciprocals of H-ion concentration.) From this it follows that below 7.0 the lower the pH the greater the intensity (or inverting power) of the acid, while above 7.0 the alkalinities *increase* with the *increase* of pH, since the hydroxyl-ion concentration increases with the increase of the pH (i.e., with the *decrease* of the H-ion concentration). Another point to be remembered is that the pH scale is a logarithmic scale, representing the powers of ten, and that therefore an acid solution of 5.0 pH, for example, would have ten times the sucrose inverting power of one of 6.0 pH while a solution of 4.0 pH would have 100 times the inverting power of the 6.0 pH solution. Similarly, a solution of 9.0 pH would have ten times the intensity of alkalinity of one of 8.0 pH, while one of 10.0 pH would be 100 times as alkaline, etc.

*Buffer Action and Buffer Solutions.*—Clark <sup>2</sup> defines buffer action as “the resistance exhibited by a solution to change in pH through the addition or loss of acid or alkali.” Buffer action is dependent on the nature and concentration of the constituents of the solution; on the nature of the acid or alkali added to the solution to effect the change in pH; and on the pH region where the buffer action is measured. The reader is referred to standard treatises on the subject for a full exposition of the theory. Sufficient to say that the solutions encountered in cane sugar factory control show buffer action to a high degree, and most of the refining materials are similarly buffered. However, solutions of pure granulated sugar show no buffer action at all while high-grade char filtered liquors are only slightly buffered.

For the colorimetric determination of H-ion concentration standard solutions of known pH called “buffer mixtures” must be employed. Buffer mixtures are buffer solutions (generally consisting of weak acids and salts of

<sup>2</sup> *Loc. cit.* p. 40.

weak acids or mixtures of both) of such definite composition that they can easily be reproduced, and whose pH value has been accurately determined by electrometric measurements. Elaborate studies by many investigators have been made in developing buffer solutions of known pH, a notable example being the series of Clark and Lubs which rise in increments of 0.2 pH from 2.0 to 10.0 pH. The solutions for making these up are now obtainable from chemical supply houses or the standard buffer solutions themselves may be purchased ready-made at reasonable rates.

**258. Colorimetric Determination of pH.—Basis of the Test.**—The colorimetric determination of pH depends upon the fact that certain indicators have a transition interval in which the color varies with changes in H-ion concentration. The test is made by comparing the color of the solution to be tested, to which has been added a known amount of a suitable indicator, with a series of buffer mixtures of known pH carrying an equal amount of the same indicator. The materials required for the tests are (1) standard solutions of known pH, (2) suitable indicators of proper strength, (3) test-tubes of clear glass of 15 mm. diameter by 120 mm. length, and (4) a color comparator to facilitate matching test solution and standards.

**Standard Solutions.**—For the preparation of the buffer mixtures the following solutions are needed; M/5 NaOH, M/5 potassium acid phosphate and M/5 boric acid and potassium chloride; all of which are best purchased ready prepared. The standard buffer mixtures from pH 5.8 to pH 9.0 are made as follows:<sup>3</sup>

COMPOSITION OF BUFFER STANDARDS AT 20° C.

pH	Milliliters, M/5 $\text{KH}_2\text{PO}_4$	Milliliters, M/5 NaOH	Milliliters, M/5 $\text{H}_3\text{BO}_3$	Milliliters, M/5 KCl
6.0	50	5.70		
6.2	50	8.60		
6.4	50	12.60		
6.6	50	17.80		
6.8	50	23.65		
7.0	50	29.63		
7.2	50	35.00		
7.4	50	39.50		
7.6	50	42.80		
7.8	.....	2.61	50.0	50.0
8.0	.....	3.97	50.0	50.0
8.2	.....	5.90	50.0	50.0
8.4	.....	8.50	50.0	50.0
8.6	.....	12.00	50.0	50.0
8.8	.....	16.30	50.0	50.0

Make to 200 milliliters

<sup>3</sup> Clark, p. 107.

The buffer solutions can conveniently be stored in 250 ml. reagent bottles with a one-hole rubber stopper carrying a 10 ml. pipette. By providing each bottle with its own pipette it is not necessary to waste solutions to rinse the pipette.<sup>4</sup>

The average laboratory will find it more satisfactory to purchase the buffer mixtures ready-made, the series ranging from 6.0 to 8.4 being those most generally needed in routine factory or refinery control.

*Indicators.*—The indicators for routine use are:

Brom-thymol blue 0.04 per cent solution, pH range..... 6.0–7.6

Phenol red.....0.02 per cent solution, pH range..... 6.8–8.4

Cresol red.....0.02 per cent solution, pH range..... 7.2–8.8

The preparation of these dyes according to Clarke follows:

Grind the amount of the pulverized dye given in an agate mortar with the number of ml. of N/20 NaOH shown.

	Gram, Dye	Milliliter, N/20 NaOH	Make to
Brom-thymol blue.....	0.4	14.1	1000 ml.
Phenol red.....	0.2	12.5	1000 ml.
Cresol red.....	0.2	11.7	1000 ml.

Schlegel and Steuber recommend storage of dye solutions and buffer mixtures in glass-stoppered Pyrex glass bottles.<sup>5</sup> They object to paraffin for sealing as ordinary paraffin has an acid reaction. Their findings as to variations in the reaction of purchased dye solutions are in accord with the experience of the present writer (G. P. M.), and the preparation of the dyes in the laboratory is recommended rather than the purchase of the ready-made solutions.

*Standard Color Tubes.*—Add 10 ml. of each of the standard buffer mixtures from 6.0 to 7.6 pH to separate test tubes after which 0.5 ml. (accurately measured) of the brom-thymol blue indicator is added to each tube. Corked and sealed with sealing wax these tubes constitute the color standards for the brom-thymol blue range. Similarly, tubes containing 10 ml. of each of the standard buffer mixtures from pH 6.8 to 8.4 are prepared and 0.5 ml. of phenol red indicator added to each to make the phenol red standards. Cresol red standards range 7.2 to 8.8 may be used instead of phenol red.

For this purpose Pyrex test tubes of 15 mm. diameter by 120 mm. length having approximately the same bore should be thoroughly cleaned and rinsed with distilled water before filling. Corks should be boiled and thoroughly rinsed before inserting. The addition of a drop of toluene to each standard to pre-

<sup>4</sup> Laboratory Methods, Cuban Sugar Club, 2d Edition, Havana, 1925.

<sup>5</sup> Ind. Eng. Chem., May, 1927.

vent mould has been recommended. Standards so prepared will last three to four weeks in routine use, or longer if protected from heat and light.

Ready-make color standards in sealed ampoules may be purchased which are fairly constant for several months if kept in a cool place but they deteriorate rather rapidly when exposed to light and routine control conditions. A set of purchased standards may be kept for reference. As will be described later non-fading glass standards are now obtainable. Standards may also be made with suitable mixtures of metallic salts.

The following mixtures have been found to match Brom-thymol blue standards very closely when viewed against "artificial daylight":

Stock Solution "A"—Blue—pH 7.6

24.6 grams  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

2.2 grams  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$

Make to 100 ml. with distilled water

Stock Solution "B"—Yellow—pH 6.0

0.45 gram  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

1.7 ml. "10 per cent solution  $\text{H}_2\text{PtCl}_6$ " (containing 2.1 grams  $\text{H}_2\text{PtCl}_6$  per 10 ml. of solution as used in the Lindo-Gladding Method for fertilizers).

Make to 100 ml. with distilled water

Intermediates are prepared from mixtures of stock solutions "A" and "B" as follows measuring from a burette.

pH	Sol. "A"	Sol. "B"	$\text{H}_2\text{O}$	pH
7.6	10.0	0	0	7.6
7.4	8.6	1.4	0	7.4
7.2	7.2	2.8	0	7.2
7.0	4.4	3.6	2.0	7.0
6.8	2.4	4.9	2.7	6.8
6.6	1.7	6.2	2.1	6.6
6.4	.9	9.1	0	6.4
6.2	.3	9.7	0	6.2
6.0	0	10.0	0	6.0

*Color Comparator.*<sup>a</sup>—A convenient comparator is shown in Fig. 99. It consists of a sliding tube rack carrying alternate tubes of distilled water and color standards and a metal frame or cover with suitably arranged apertures for comparing the colors. Immediately in front of the sliding rack are sockets for two removable tubes for the solution to be tested, spaced the same distance as the tubes in the rack. Ordinary daylight, or any of the artificial daylight lamps serve as a light source, the simplest arrangement being to place a white

<sup>a</sup> Meade and Baus. The Planter, June 27, 1925.

surface at 45° behind the apertures and hang a shaded Mazda "daylite" globe above this.

*Procedure of Test.*—Test tubes of the same bore as those used for the color standards but 150 mm. long are used for the test solutions and those tubes for convenience should be marked for 10 ml. (marking in laboratory with "Diamond Ink"), 15 ml. and 20 ml. If the solution to be tested is not to be diluted, 10 ml. is measured into each of two tubes, 0.5 ml. (accurately measured) of indicator added to one of these and the dye and solution mixed by rotating the tube or by a stirring rod or by closing the tube with a clean cork and shaking. Under no circumstances should the tube be closed with the finger or hand because of the adverse effect of the acid perspiration. The tube containing the dye solution is placed before the left-hand aperture, the blank before the other aperture and the rack moved back and forth until the standard to match the test sample is behind the blank solution. Thus any color and turbidity in the blank solution is added to the color of the standard and compensates for the color and turbidity in the test solution, while the tube of distilled water behind the test solution compensates for the water in the blank.<sup>7</sup>

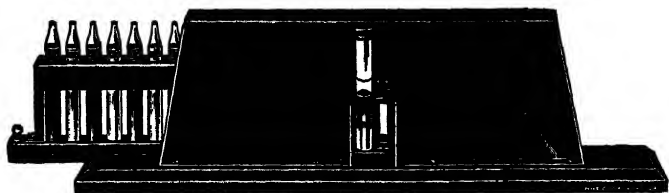


FIG. 99.—Color Comparator for pH Determinations.

Workmen of ordinary intelligence can be trained to make a test with this comparator to 0.1 pH.

Most investigators say that dilution has little or no effect on the pH of all but the purest solutions encountered in sugar work (*e.g.*, high-grade refinery liquors and solutions of granulated sugar) while others show that dilution is to be avoided. For colorimetric purposes the best practice lies somewhere between these two extremes. A moderate dilution (1 : 3 or 1 : 5) with distilled water has not been found to affect the results in such solutions as washed sugar liquor or pan house sirups in the refinery or of limed and clarified juice, sirup (*meladura*) and press juice in the cane house. This dilution minimizes the disturbing effect of color and turbidity, and adherence to a fixed dilution gives comparable results at all times. The use of kieselguhr and filter papers should be avoided. Filter-Cel is acid and "Hy-Flo" Filter-Cel alkaline, while all ordinary laboratory filter papers are acid.

A 1 : 3 dilution can easily be effected in routine control by having test tubes marked at 10, 15 and 20 ml. Distilled water is added to the 15-ml. mark then the sugar solution to 20 ml. and the two mixed by pouring from one tube to another. Half the mixture is then poured back in the first tube, 0.5 ml. of

<sup>7</sup> Walpole, *Biochem.* Jan. 5, 207 (1910).

indicator dye added and the comparison made as before. The measurement of the 0.5 ml. of indicator solution can conveniently be carried out by the Koch measuring pipette arranged as shown in Fig. 100. The soda-lime tube attachment prevents contamination by the  $\text{CO}_2$  of the air. Solutions should be tested at or near room temperature as the pH is quite markedly affected by differences in temperature.

A further simplification of the above procedure has been devised by the writer (G. P. M.) for use by the blowupsmen in refinery control. Phenol red dye is diluted with neutral water until each 7.5 ml. contains 0.5 ml. of the standard dyer (66.6 ml. phenol red 0.02 per cent : 1000 ml. distilled water.)



FIG. 100.  
Koch Pipette  
for pH Dyes.

Test tubes marked at 7.5 ml. are used, to one of which is added the diluted dye, to the other any clean tap water; after which a 2.5 ml. (approximately) measure of material to be tested is added to each tube. Clean corks are placed in the tubes, the solutions shaken and the readings taken in the comparator as before. This method does away with the use of neutral water and with the necessity of accurate measurement of the dye by unskilled workmen and reduces possibility of contamination to a minimum. Tap water is used for rinsing tubes and corks. The diluted dye keeps for two or three days but is best made up daily in the laboratory. Constant checks by regular laboratory methods have proved this simplification to be thoroughly reliable.

*Spot Test.*—As described by Brewster and Raines<sup>8</sup> "this consists of adding one drop of the appropriate indicator solution to three drops of the test liquid contained in the depression of a porcelain spot plate. The color developed in the test spot is matched with similar spots made by adding a drop of the same indicator to three drops of a standard buffer solution. The pH of the standard buffer which matches the test solution in hue is the same as the pH of the test solution."

This method is no longer in general use as it has no advantages over the color comparator methods using fixed standards. The disadvantages are obvious, the greatest of which, namely the necessity of making "spot" standards for each test has been overcome by the Hawaiian Sugar Planters Association by the adoption of spot color standards made by means of dyes on celluloid.<sup>9</sup> This has the advantage of doing away entirely with standard buffer solutions, but it does not compensate for the color and turbidity of the test solution. Cook<sup>10</sup> later reported on a comparison between a thousand tests by this method and by potentiometer and showed that in general agreement was within 0.2 pH, more reliable results being obtained with dilution of juice 1 : 3 than on undiluted juice. Some wide differences were noted, particularly in the upper

<sup>8</sup> The Planter. Sept. 9, 1922.

<sup>9</sup> H. A. Cook. Facts About Sugar, June 20, 1925.

<sup>10</sup> Int. Sug. Jour. Vol. 29 (1927), No. 348.

range of brom-thymol blue, but these were obviated by using phenol red and cresol red for pH 7.2 to 7.6.

*Test Papers.*—In Europe test papers made of cresol purple, bromthymol blue, coralling and phenol red have been indorsed by Prinsen Geerligs and Dr. Herzfeld<sup>11</sup> as being simpler than American colorimetric practice, but these have not found favor on this side of the water.

*Helige Comparator with Glass Color Standards.*—This is shown in Fig. 101. Each indicator dye has a separate color disk similar to the one shown to the right of the comparator, in which are arranged eight or nine permanent glass standards made from solid glass, non-fading. The test is made as described before using a blank in one of the square tubes and the test solution with 0.5 ml. of dye in the other. The proper color disk, inserted in the instrument, is rotated until the color of the glass and the test solution match, when the pH is read in the small aperture on the right front face of the comparator. To facilitate matching a prism is placed in the eyepiece which brings the two colors together in the halves of a circle, similar to the field of a polariscope.

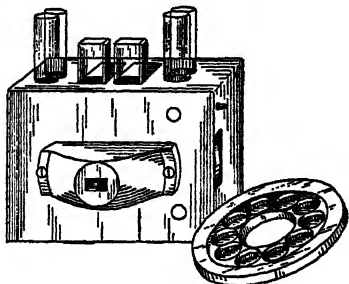


FIG. 101.—Helige pH Comparator.

Care must be taken to use only correct daylight (either real or artificial) for this instrument; otherwise the color disks will not match the dye solutions.

*"Neutral" Water or "Dawson" Water.*—Distilled water is always acid, ranging from 6.0 to 6.4 pH depending upon how recently it has been boiled. The pH is so sensitive to absorbed CO<sub>2</sub> that a drop in pH of several tenths may be effected by pouring recently boiled distilled water from one test-tube to another.

So-called "Dawson" water<sup>12</sup> or "Neutral" water may be made by double distilling water and then boiling off one-third of the volume after which it is stored in Pyrex containers, fitted with a soda-lime tube or kept from contact with air. Schlegel and Steuber<sup>13</sup> found this water to be a reliable reference standard for the preparation of bromthymol blue solutions. The water will not keep its neutral character in ordinary routine determinations. An ordinary distilled water is satisfactory for dilutions for most sugar-house and refinery materials.

*Sources of Error in Colorimetric Determinations.*—Certain obvious errors are to be avoided such as the use of tubes of different bores; inaccurate measurement of indicator dye; working with hot solutions; contamination of tubes or solutions by contact with hands; failure to rinse tubes before making

<sup>11</sup> Int. Sug. Jour. Vol. 27 (1925), No. 317.

<sup>12</sup> L. E. Dawson, Jour. Phys. Chem. Vol. 27 (1921), 551.

<sup>13</sup> Loc. cit.

test. The greatest possibility of error is in the use of color standards that have faded because of exposure to light, heat or air.

Much greater precautions must be exercised in testing unbuffered or slightly buffered solutions. Here exposure of test solutions to  $\text{CO}_2$  of the air, minor variations of reaction of dye solution itself; use of other than "neutral" water for dissolving or diluting; and excessive dilution, may all cause such wide variations as to vitiate the value of the test completely.

**259. Electrometric Determinations.**—Electrometric methods of determining pH using the potentiometer are somewhat complicated for use in routine work, though they will always be the basis of standardization for the other methods. Balch and Paine<sup>14</sup> speak of the hydrogen electrode as "a tool for the research chemist." The quinhydrone electrode is of simpler technique and its use in automatic recording of pH is described elsewhere (40). Several complete potentiometric outfits are on the market, the one shown in Fig. 102 being made by Leeds and Northrup.<sup>15</sup> This indicator

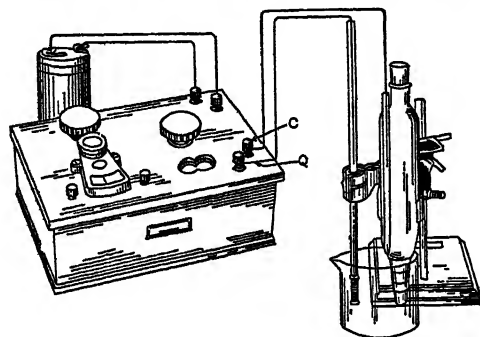


Fig. 102.—Electrometric pH Apparatus.

consists of two essential parts—the standardizing system and the operating system. The standardizing system consists of a Wheatstone bridge in one arm of which the standard is connected, another arm contains a fixed resistance, while the third and fourth arms are formed by a variable resistance. The null point indicating galvanometer of a highly sensitive type, is bridged across the system from the swinging arm of the resistance to the contact point between the standard cell and the fixed resistance. Power for operating the bridge is derived from a single dry cell battery of about 1 to  $1\frac{1}{2}$  volts.

The operating system is similar to the standardizing system; in fact, it is the same system except that a special quinhydrone cell is substituted for the standard cell. The quinhydrone cell has two parts. One part is a standard calomel half cell, composed of a platinum electrode dipping into a layer of mercury in contact with calomel and saturated potassium chloride. The other part is a gold electrode. Both electrodes can be mounted on a regular laboratory support so they dip into the unknown solution.

To standardize the potentiometer, press the plug switch located just to the left of the galvanometer scale. This closes the Wheatstone bridge circuit through the standard cell which is mounted within the box. The needle of the galvanometer is brought back to zero by rotating the knurled knob which controls the variable resistance, after which operation the plug switch is released.

<sup>14</sup> The Planter. October 31, 1925.

<sup>15</sup> From description supplied by makers.



The potentiometer is ready for a determination and need not be reset, except for an occasional check. To make a pH determination of an unknown solution, the calomel cell and electrode are mounted on an apparatus support so that the ground glass and gold electrode are completely immersed in the liquid in a small beaker. A wire is run from the binding post, (marked *Q*) at the front of the box and dipped into the mercury of the gold electrode. Another wire is run from binding post (marked *C*) to the calomel cell. Quinhydrone is then stirred into the solution until saturated. However, as it is very slightly soluble, only a small quantity is necessary.

The solution is then stirred and the plug switch, just at the right of the galvanometer scale, is pressed, closing the bridge through the quinhydrone-calomel cell circuit. The needle will then swing to the right or left, depending on the hydrogen ion concentration of the solution. The knurled knob on the right is then rotated until the needle returns to zero, and the reading noted on the scale just in front of it. The setting should be checked several times as it takes about one-half minute for the system to come to equilibrium. Take the solution temperature with a mercury thermometer and with these data, read directly the pH value from the graph curves supplied with the instrument.

The approximate pH range of the *L* and *N* Indicator is 1 to 9. Above a pH of 9 the hydrogen electrode should be substituted for the quinhydrone electrode.

stirred. When the asbestos has partly settled a slow stream of water is allowed to flow upward through it to carry off the fine, almost colloidal material and leave the fiber. The vessel is placed in a sink and the flow allowed to continue about forty-eight hours. Very little attention is required once the flow is regulated so as not to carry off an excessive amount of fiber. A few times a day the flow is stopped and the mixture stirred to loosen the fine material. After settling somewhat the flow is again started.

At the end of the washing period, most of the water is removed by suction through an inverted filter made by tying a piece of fine cloth over a Gooch adapter or other form of tubing and connecting with the vacuum pump. The asbestos is finally poured on a Buchner funnel under vacuum and the excess water pressed out with a pestle or flat glass stopper. It is then digested twenty-four hours on the steam bath with 2 liters distilled water, 200 cc. strong HCl and 70 cc. strong HNO<sub>3</sub>. The now perfectly white fiber is filtered on the Buchner again and washed 8 or 10 times with boiling distilled water, an effort being made to drain off as much as possible at each washing by suction and pressing upon the pad. It is easy to get rid of the acid. The fiber is dried at any temperature from 100 to 120° C. and bottled for use.

*Preparation of Optical Filtrates.*—More stable optical filtrates are obtainable if the final solid content is 60 to 65 Brix instead of 55 Brix as recommended by Peters and Phelps. In the case of sugars a quantity is weighed approximately to furnish the desired volume at the desired Brix. For example, if 250 grams of solution are desired, 150 grams of sugar are weighed, transferred to an Erlenmeyer flask and the whole (with stirring rod) roughly tared and partially immersed in water at 65°–70° C. Boiling distilled water is added to the sample in small quantities at a time with thorough mixing. The temperature and the mixing hasten solution and at the same time a high Brix is maintained. This is the essential feature of solution since it appears that high sugar concentration stabilizes the colloids and prevents their coagulation later. In other words, sugar at high concentration acts like a protective colloid in preserving a high degree of dispersion. When all is dissolved the desired Brix may be obtained by placing the tared flask and contents on the rough balance and adding hot water in small lots with continuous stirring until the desired water weight is reached. The sirup is cooled and is ready for filtering. To 250 grams sirup we add 1 gram of "A" asbestos and shake violently five minutes. Two filtering pads are prepared. Use 60 cc. Jena glass filtering crucibles marked 2-G, 3, with adapters in place of the Gooches and bolting silk formerly recommended. For the first filtration shake up 0.5 gram "A" asbestos with water and form an even pad and 1.0 gram of XXX asbestos is used for the final pad. A little of the sirup mixed with the "A" grade asbestos is poured on the "A" pad and drawn through to displace the water, when a dry clean receiver is put in to take the main sample. The same procedure is employed when the "A" filtrate is formed on the XXX filter, the object being to prevent dilution. The "A" pads are discarded after use unless several samples during the day are to be filtered in which case they may be washed with hot water and used again. The XXX pads are washed with hot water and sucked fairly dry after which they are placed in a flask containing the dilute aqua regia mentioned above. When enough have accumulated they are digested and the asbestos used again. Pads should not be allowed to dry on the glass filtering element. When this occurs the asbestos becomes very adhesive and forms a coating on the surface of the glass that is almost impervious to water.

Peters and Phelps in Tech. Paper 338 stress the importance of cleanliness of apparatus to receive optical filtrates. Treat Erlenmeyers, filters, bottles and glass cells with chromic-sulfuric acid cleaning mixture after use and since finally with distilled water before drying.

*Effect of pH on Color.*—The effect of a change of H-Ion concentration on the color of a sugar solution has been studied by many investigators. It has

long been known that acid juices and sirups are lighter colored than the same juices at alkaline reactions, the coloring matter of the juice or sirup acting as a sort of indicator. The light absorption at different wavelengths also differs markedly as the pH is changed. Zerban<sup>7</sup> showed that using a raw sugar solution which had been altered from 5.8 pH to 8.3 pH through small intervals by the addition of NaOH there was an appreciable increase in the  $-\log t$  values (measure of the amount of color) with the increase of the pH, though within the range of pH of refinery work (pH 6.6-7.9) the increase is equivalent to a concentration of the same coloring matter, the quality not changing appreciably.

In all comparative color work the pH of the solutions compared should be accurately adjusted to the same value in order to avoid this effect of the change of pH.

*Kieselguhr for Routine Color Work.*—While it is admitted that kieselguhr has a selective action on the coloring matter, its convenience for routine work as compared with the tedious method of Peters-Phelps and Brewster is evident. There is no publication available showing quantitatively the difference between kieselguhr and asbestos filtration. The work of Zerban<sup>8</sup> and of Peters and Phelps<sup>9</sup> shows the difference between asbestos filtration on *concentrated* solutions and kieselguhr on *dilute* solutions, the latter invariably showing about 20 per cent more color than the former but the variation ranging from +2.43 per cent to +36.64 per cent so that no correction can be applied to convert the results of one procedure into those of the other. These variations are the result of two errors, the dilution and the selective action of the kieselguhr. Peters and Phelps<sup>10</sup> promised analytical proofs in a later publication of the difference due to kieselguhr alone, but these have never been published.

In view of the divergence of opinion on asbestos filtration and the tediousness of the procedure, it appears that the use of kieselguhr for routine color measurement must be continued pending other simple methods of greater accuracy. The solution methods advocated by the Bureau of Standards, avoiding dilution, can be used to obviate part of the error shown by Peters and Phelps and Zerban, and the routine should be standardized as to quantity and class of kieselguhr used, freedom from turbidity, density of solution, etc. It is understood, of course, that for spectrophotometric work the asbestos filtration must be used.

#### APPARATUS FOR COLOR MEASUREMENT

The color instruments described here are those most commonly used in American sugar practice. There are a great many varieties of colorimetric apparatus but practically all of them operate on one or another of the principles illustrated by the instruments given below.

<sup>7</sup> Unpublished paper presented before A. C. S. Meeting, 1925.

<sup>8</sup> Unpublished. Presented before Am. Chem. Soc., 1925.

<sup>9</sup> B. S. Paper. No. 338, p. 294.

<sup>10</sup> *Loc. cit.*, p. 283.

**262. Colorimeters.—Duboscq Type.**—The simplest method of comparing the amount or intensity of color of two liquids is by adjusting the depth of the liquids in separate tubes until they appear to have the same color as viewed from above. The Duboscq type of colorimeter, modifications of which are made by many manufacturers of optical instruments, is designed to accomplish this adjustment and comparison. The one shown in Fig. 103 is a Klett-Kober Duboscq instrument. Light falling on the adjustable porcelain reflecting plates (mirrors are used in many other instruments) is projected up through the clear glass bottom of the black glass tube containing the liquid to be examined. A black glass plunger, also with transparent polished glass bottom, is immersed in the liquid and transmits the light passing through the liquid,

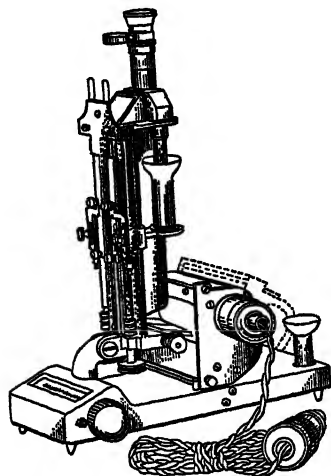


FIG. 103.—Klett-Kober Colorimeter.

between the bottom of the tube and the bottom of the plunger, to the prism above and thence to one-half the field in the eyepiece. An exactly similar tube on the other side of the instrument carries the liquid used as a standard, the light transmitted through it forming the other half of the field. Either tube may be raised or lowered by the milled heads, thereby changing the depth of liquid through which the light passes, until the two halves of the field appear the same color. By means of the small telescope attached to the rear of the eye-piece the scales showing the depth of liquid below the plunger in each tube may be read to fractions of a millimeter. In practice the standard is generally placed at some convenient fixed depth (say 20 mm.) and the test solution adjusted to match it.

Then the amount of color in the test solution in terms of the amount of color in the standard is computed by dividing 20 (or whatever depth was chosen for the standard) by the depth of the test solution. A convenient adjunct to the Klett-Kober is the table in the base of the instrument by which this calculation is effected.

The Duboscq type of colorimeter is simple and convenient, and accurate for the purpose intended (that is, measurement of total color), so long as the standard and the test solution are of identically the same color. When differences in tint have to be dealt with, which is the case in dealing with filtered and unfiltered sugar liquors, there is considerable difficulty in matching the shade. The standards generally used in sugar-work are caramel in glycerine, or ferric chloride solutions, and while these solutions approximate the color of washed sugar liquor or solutions of raw sugar they differ markedly in tint from char-filtered liquors. A little ferric chloride added to the glycerine-caramel standards (suggested by T. B. Wayne) adds to the simulation of the color of the washed sugar liquor but there are still differences in tint to be dealt with.

These differences can be removed to some extent by inserting a light filter in the colorimeter between the solution and the eye. In the Klett-Kober this is easily accomplished by adding to the hollow plungers (which may be unscrewed) the following solution of metallic salts to a depth of 1 or 2 cm. (Obviously both plungers must contain the same depth of solution.) The solution which was devised by Wayne<sup>11</sup> consists of 250 ml. of 10 per cent nickel nitrate and 5.65 ml. of 10 per cent potassium bichromate (both solutions having been filtered through asbestos as described on p. 235) made to exactly 500 ml. with distilled water. It has an effective wavelength of 563  $m\mu$  and transmits only a narrow range of wavelengths between 540 and 570  $m\mu$ , and when used in proper thickness, the effect is to filter out the rays above and below this range and remove the differences of tint.

The standards used in the above method may be calibrated to absolute units (see page 295) in a spectrophotometer or an arbitrary standard may be used. The use of a standard that will give comparable results with other workers is an argument for the "standardized" standard. Kenneth Ritchie<sup>12</sup> gives a method of making a standard of ferric chloride solution which can be translated into Stammer units (see following paragraph).

*Stammer Colorimeter.*—This is one of the oldest of the colorimeters and is still used extensively in Europe, although being rapidly replaced by color analysis apparatus. In effect it is similar to a Duboscq in which the standard tube has been replaced by two brown colored glasses, accurately standardized, called "normal glasses." In the Stammer the plunger is moved up and down, instead of the tube, to vary the depth of the test solution.

The reference basis is that a solution 10 c.m. high at 100° Brix equal in color to two normal glasses has a color of 1° Stammer. Stammer degrees are actually calculated by dividing 100 by the height in millimeters of test liquid which matched the color of two normal glasses and then multiplying this by 100/Brix  $\times$  Sp. Gr.

The Stammer offers even greater objections than the Duboscq on the score of tint differences between standard and solution. The normal glasses were made for beet juices and do not match cane-sugar products well in tint. It has the advantage that it gives results in units which are comparable with the work of other Stammer instruments.

*Colorimeters Depending on Photo-electric Cells.*—Recently several instruments have been devised in Europe for measuring color depth (or rather extinction by colored solutions) that do away with the human eye entirely. One described by K. Sandera<sup>13</sup> is "the apparatus schematically represented in the accompanying diagram (Fig. 104.) The principle employed is measurement of the strengths of electric currents generated by light of varying intensities.

"In this apparatus light from the source, *O*, passes through a suitable lens, *e*, and filter, *f*, a cuvette, *a*, containing the pure solvent and impinging on the coating, *K*, of the metallic potassium on the interior of the photo-electric

<sup>11</sup> The Planter. October 17, 1925.

<sup>12</sup> Ind. Eng. Chem. Vol. 19 (1927), No. 11.

<sup>13</sup> Zeit. Zuckerind. Czech. Rep. Vol. 52 (1928), p. 261.

cell. The photo-electric current is amplified by the electron tube (grid,  $M'$  and anode,  $E$ ); the amplified current, which is proportional to the light falling on the photo-electric cell, is measured on the millivoltmeter,  $MV$ . The cuvette  $b$ , containing a solution of the product, is then put in place of  $a$  and a new measurement made; the difference in the two measurements gives the measure of the color of the sample. The accuracy of the readings is about 1 per cent. The instrument is made by J. & J. Fric, Prague."

**263. Instruments for Color Analysis or Tintometry.—*Lovibond Tintometer.***

—This is one of the oldest instruments for analyzing color. It consists of a trough carrying the liquid to be examined, at the side of which is a tube with slots for the standard color glasses, which are of the primary colors, red, yellow and blue, with numerous glasses of varying depths of color each accurately

graded and numbered.

The operator "builds up" the color of the test solution by super-imposing the correct amount of red, yellow and blue glasses. The results are then expressed in units of red, yellow and blue by adding together the numbers on each of the glasses of the different colors used. It is obvious that the operation requires a delicate color sense and is not suited to routine operations. The color glasses are expensive and where a wide

range of them are needed as in sugar work the cost is a

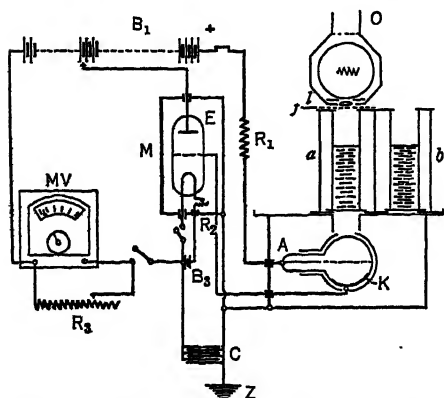


FIG. 104.—Colorimeter Using Photo-Electric Cell.

factor. The Lovibond has been largely superseded by newer models of color analyzing instruments.

***Ives Tint-Photometer.***—This is generally known as the "Hess-Ives" because the first model was so named. Although not a true spectrophotometer it is a step in that direction as it measures the amount of light transmitted by the test solution as viewed through a series of color screens or light filters each of which isolates a part of the spectrum. It may be used for determining the color of solids also, and a description of the method of using it for soft-sugar gradings, as devised by Dr. J. W. Schlegel, has been described by L. A. Wills.<sup>14</sup>

The optical arrangement as shown in Fig. 105 is such that a part of the light from the lamp is reflected from the 2d magnesia block to the 2d mirror, thence through the glass cell through one-half of the lens  $K$ , forming one-half of the field; while a second part of the light passes first through the adjustable slit, thence through condensing lenses to the first magnesia block where it is reflected to the first mirror and thence through the other half of  $K$  to form the remaining half of the field.

<sup>14</sup> Facts About Sugar. Nov. 20, 1926.

For liquids, the glass cell is first filled with the pure solvent (water) and put in position while the lever *L* is placed at 100 (i.e., slit is wide open) on the scale *S*. With one of the color screens in place, the 2d magnesia block is adjusted by means of the lever *LL* until the field appears evenly colored as viewed from the eyepiece. The water cell is then replaced by a similar one containing the test solution. The field will now appear darker on one side due to the extinction of light by the color of the solution. This is compensated for by closing the slit with the lever *L* until the two halves of the field again match, when the reading is noted on the scale. In a similar manner, readings are taken through each of the color screens. There are five of these provided with the instrument; blue, blue-green, green, yellow-green and red. Honig and Bogtstra<sup>15</sup> have subjected these color screens to spectrophotometric analysis to find the maximum wavelength and the spectral range transmitted. They found that combinations of these light filters greatly reduced the range of transmission; their results being given below:

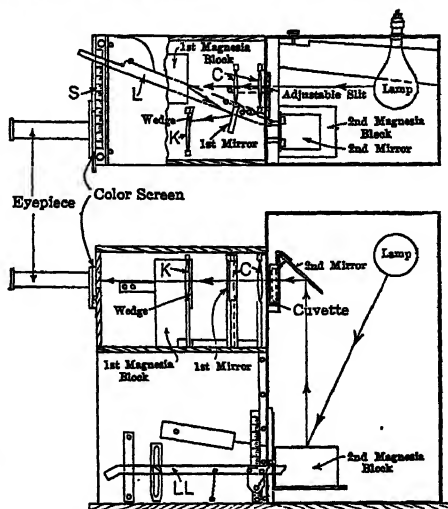


FIG. 105.—Ives Tint-Photometer.

Simple Filters	Maximum Transmission at	Transmitted Traject in $m\mu$
Blue.....	465 $m\mu$	60 (420–480)
Blue-green.....	500	40
Green.....	535	60
Yellow-green.....	575	40 (565–605)
Red.....	625	70
Combinations:		
Blue-green-green.....	512	10
2 X green.....	535	50
Yellow-green + green.....	560	10
2 X yellow-green.....	575	20
Yellow-green + red.....	617	20

<sup>15</sup> *I., op. cit.*

A "560" color screen having a maximum transmission of 560  $m\mu$  is now made for this instrument also.

In the early days of the Hess-Ives an investigation by the writer and Joseph B. Harris<sup>18</sup> showed that the percentage scale on this instrument was meaningless in itself but that the relationship between the scale readings and relative amounts of color was expressed algebraically as  $Y = K^x$  where  $y$  is any scale reading,  $K$  the reading for any unit of material and  $x$  the number of units of material required to give the scale reading  $y$ ; or  $x = \log y / \log K$ . To avoid the logarithmic calculation a table was devised as given below by taking  $K$  as 99 and solving throughout for  $y$  from 100 to 0.

TABLE FOR TRANSLATING HESS-IVES TINT-PHOTOMETER SCALE READINGS TO MEADE-HARRIS UNITS OF COLOR

$y = K^x$ , where  $y$  = scale reading;  $x$  = units of color;  $K = 99$  (constant)

Scale Read- ing. $y$	Units of Color. $x$	Scale Read- ing. $y$	Units of Color. $x$	Scale Read- ing. $y$	Units of Color. $x$	Scale Read- ing. $y$	Units of Color. $x$	Scale Read- ing. $y$	Units of Color. $x$
100	0.0	80	22.2	60	50.8	40	91.1	20	160.0
99	1.0	79	23.4	59	52.5	39	93.6	19	165.0
98	2.0	78	24.7	58	54.2	38	96.2	18	170.5
97	3.0	77	26.0	57	55.9	37	98.9	17	176.0
96	4.0	76	27.3	56	57.7	36	101.6	16	182.0
95	5.1	75	28.6	55	59.4	35	104.4	15	189.0
94	6.1	74	29.9	54	61.3	34	107.3	14	196.0
93	7.2	73	31.2	53	63.1	33	110.3	13	203.0
92	8.3	72	32.6	52	65.0	32	113.3	12	211.0
91	9.4	71	34.0	51	67.0	31	116.5	11	219.0
90	10.5	70	35.5	50	68.9	30	119.7	10	229.0
89	11.6	69	37.0	49	70.9	29	123.1	9	239.5
88	12.7	68	38.4	48	73.0	28	126.6	8	251.0
87	13.8	67	39.9	47	75.1	27	130.2	7	264.0
86	15.0	66	41.3	46	77.2	26	134.0	6	280.0
85	16.2	65	42.8	45	79.4	25	137.8	5	298.0
84	17.4	64	44.4	44	81.6	24	141.9	4	320.0
83	18.6	63	46.0	43	83.9	23	146.2	3	349.0
82	19.8	62	47.6	42	86.2	22	150.6	2	389.0
81	21.0	61	49.2	41	88.7	21	155.0	1	458.0

These units proved convenient for several years until the absolute units recommended by Peters and Phelps (to which the above units may be translated) came into vogue, and they are still of service in routine work. In 1924

<sup>18</sup> Meade and Harris. Ind. Eng. Chem., 12 (1920), p. 686.



E. W. Rice devised a scale for the Hess-Ives based on reciprocals of these units.<sup>17</sup> Later Rice made a scale for the "560" color screen which reads in  $-\log t$  values.

The Hess-Ives tint photometer may be read accurately to 1 division of the scale with solutions of moderate color concentration (*e.g.*, washed sugar liquor in a 2 cm. cell) but with more difficulty through the blue screen. With greater color concentrations cells of 1 cm. thickness may be used or the test solution may be diluted with the clear sucrose sirup described on p. 285 (never with water). This instrument is comparatively inexpensive, will serve for routine work, and is valuable for many kinds of investigations though it is hardly of the high accuracy necessary for research.

*Keuffel and Esser Color Analyzer.*—This is a direct-reading spectrophotometer of the highest optical precision suitable for all classes of research work

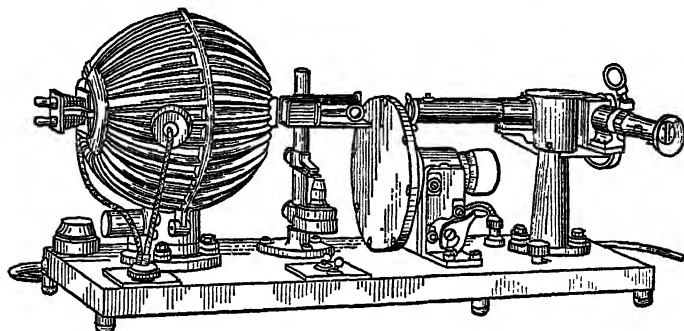


FIG. 106.—K. & E. Color Analyzer.

and color investigations both with solids and liquids. It consists essentially of (1) a source of light which consists of two 400-watt lamps in the spherical housing at the left of Fig. 106 (2) a rotating disk photometer and (3) a constant deviation spectrometer. Between the lamp-housing and the disk of the photometer is seen the holder for liquid samples or transparent solids. Opaque samples are held at the rear of the lamp-housing by the spring clips seen at the left of the figure. Full directions for the operation of the instrument are supplied by the makers.

Because of its complexity and cost the K. & E. analyzer is not suited for general routine work, but most of the recent American investigations of color in sugar products have been carried out with this instrument.

**264. Color Nomenclature for Sugar Solutions.**—The symbols used here are those adopted by the Bureau of Standards following the recommendation of the Optical Society of America:<sup>18</sup>

<sup>17</sup> Planter. Nov. 15, 1924.

<sup>18</sup> See Peters and Phelps Bur. St. Tech. Paper 338, Part I, 3 for a more detailed discussion.

$T$  = transmittancy, or the fraction of the incident light which is transmitted through a vessel with parallel polished glass ends containing the colored solution, after correcting for reflection at the surfaces and for absorption of light if any by the pure solvent. The solvent for sugar solutions is pure water which has an absorption which is negligible.

$t$  = specific transmissivity = transmittancy reduced to unit conditions of thickness and concentration.

$b$  = thickness in centimeters of the absorbing solution.

$c$  = concentration in grams original colored dry substance (sucrose + non-sugars + coloring matter) per 1 cc. of solution.

Lambert's law states that the amount of light transmitted when the thickness of the absorbing medium is varied (concentration remaining the same) is inversely proportional to the power of the units of thickness or

$$t = \sqrt[b]{T}.$$

Similarly Beer's law is that the light transmitted varies inversely as the power of the concentration (thickness remaining the same) or

$$t = \sqrt[c]{T}.$$

Combining these the Lambert's-Beer's law becomes

$$t = \sqrt[cb]{T} \quad \text{or} \quad -\log t = \frac{1}{cb} (-\log T)$$

and  $-\log t$  is a measure of the coloring power (i.e., intensity of absorption) of the unknown amount of coloring materials associated with 1 gram of saccharine dry substance.

$A$  = specific absorption =  $1 - t$ ,

$\log A = \log (1 - t) = -\log t$

$-\log t$  = specific absorptive index.

Peters and Phelps found that for the ordinary coloring matter of sugar products the solutions appear brightest to the eye at the wavelength ( $\lambda$ ) of 560 millimicrons ( $m\mu$ ) or expressed in spectrophotometric terms

$\lambda = 560 m\mu$  is the wavelength of the optical center of gravity of the luminosity curve for unit quantity of coloring matter of sugar products.

It is for this reason that the wavelength of 560  $m\mu$  was chosen as a standard by the Bureau of Standards. The Europeans use 590  $m\mu$  (Lunden) and 610  $m\mu$  (Spengler and Landt).

$Q$  = Absorption ratio = the specific absorptive index at any one wavelength compared with the specific absorption index at the standard

$$\text{wavelength of } 560 = \frac{-\log t_{\lambda}}{-\log t_{560}}.$$

$Q$  is a measure of the quality of absorption of a solution; it is the relationship between unit absorption at 560  $m\mu$  and absorption at other wavelengths

throughout the spectrum. A series of  $Q$  ratios taken at definite intervals of wavelengths and plotted as a curve will show the "color trend" of the solution being studied while comparisons of the  $Q$  ratio curves of two solutions (e.g., before and after char filtration) will show the differences in the kind of color in the two solutions studied.

Peters and Phelps also give a calculation for color units  $n$ .

$$n = \frac{-\log t_{560}}{.00485}.$$

These color units do not seem to have come into as general use as the  $-\log t$  values themselves.

*Determination of  $-\log t$  Value in Practice.*<sup>19</sup>—After a suitably transparent solution has been prepared with a correct dry substance concentration and a suitable color concentration a parallel-sided cell of suitable thickness is filled and placed in one beam of a photometer. An exactly similar cell filled with distilled water or with a colorless sucrose solution is placed in the comparison beam in order to compensate for losses by reflection at the cell surfaces and eliminate corrections for this effect. A spectrophotometer, a simplified spectrophotometer, or other color measuring device which is capable of measuring transmittancy for monochromatic light of wavelength  $560 m\mu$ , or for a narrow band of the spectrum whose effective wavelength is  $560 m\mu$ , may be utilized. If a mercury vapor lamp is used as a light source measurements of  $T$ , or  $-\log T$ , are made for the green line,  $546 m\mu$ , and the yellow lines,  $578 m\mu$ . The reading for  $560$  is then obtained by deducting 48 per cent of the difference between  $-\log t$  at  $\lambda 546$  from  $-\log t$  at  $\lambda 578$ ; the result is  $-\log t$  at  $\lambda 560$ .

The transmittancy measurement  $T$  determined for the concentration,  $c$ , and thickness,  $b$ , is reduced to unit basis, as regards concentration and thickness, by means of the equation  $t = \sqrt[cb]{T}$ , or  $-\log t = \frac{-\log T}{cb}$ , which expresses Lambert's-Beer's law. It is to be noted that the photometer scale may be graduated in terms of  $T$  (transmittance), or directly in  $-\log T$ , the latter direct reading eliminating one step in the calculation. The thickness,  $b$ , for any cell is a constant, and  $c$  is readily calculated, utilizing for this purpose the refractometric and density measurements.

In all cases, whether or not the colored solution has been diluted, the concentration,  $c$ , is expressed as grams of original colored dry substance per 1 cc. of the final solution upon which the transmittancy measurement is made.

<sup>19</sup> Peters and Phelps, *Loc. cit.*, p. 283.

## CHAPTER XXIV

### SAMPLING AND AVERAGING

**265. General Remarks on Sampling and Averaging.**—One of the most difficult, and often unsatisfactory, problems for the cane-sugar chemist is that of securing representative samples of the juices and various products at the different stages of the manufacture. If a sample does not strictly represent the average composition of the material, the analytical work will usually be of but little if any value.

In order that the samples may be representative they must be drawn continuously in proportion to the quantity of the material. Or, they must be secured at intervals, drawing a definite quantity in each sample, from a measured or weighed quantity of the material, the size of the sample always bearing the same relation to the amount of material sampled. This second method is the one usually practiced and is termed sampling by aliquot parts.

The importance of a proper method of sampling is illustrated by the following example: Given four lots of sirup *A*, *B*, *C*, and *D* from which an average sample is to be drawn. Let  $A = 1000$ ,  $B = 800$ ,  $C = 500$ , and  $D = 200$ , and let each lot differ from the others in analysis. Manifestly a mixture of equal parts of sirup from these lots would not be a true average sample, but a mixture of 10 parts of *A*, 8 of *B*, 5 of *C*, and 2 parts of *D* would represent the average composition of the sirup.

In averaging the analyses of the various materials in a cane-sugar house, it is advisable to use the weights rather than the volume. Thus the weights of the juice, the sucrose, and the apparent solids (Brix) should be recorded daily and at the end of a "run" or period the sum of the daily weights of sucrose divided by the weight of the juice and the quotient multiplied by 100, will give the mean per cent of sucrose in the juice and so on. Similarly the analyses of the sugars, and, so far as possible, those of the other products should be averaged.

For the general laboratory data, it is preferable to collect daily one composite sample of the juices and each product. This is advisable since the quantity of material represented by the analyses is usually known or estimated daily, thus giving the analytical work a definite value.

**266. Sampling the Cane in the Field.**—It is practically impossible to secure a moderate-sized sample of sugar-cane that will be even fairly representative of that of a field. The best the chemist may hope to accomplish, under favorable conditions, is to obtain a sample that will in a very general way indicate the condition of the cane. The difficulty in sampling is due to the great variations in the analysis of canes from the same stool and also from various parts

of even a small field. Frequently in a large factory, receiving cane from many fields, the daily average analyses will differ but little from day to day whereas single analyses may vary widely from the average.

In sampling cut cane a few stalks should be selected from every second or third row, crossing the field one or more times, according to its size, in sampling. The large sample, after mixing the canes, should be reduced by sub-sampling to one of convenient size for the laboratory. This method is frequently impracticable, since the carts often follow close behind the cutters.

It is even more difficult to sample standing cane, since the field is an almost impenetrable jungle. In this case a few entire stools of cane should be secured from various parts of the fields, but not from near ditches or headlands, and these canes should be reduced to a convenient number, after mixing them thoroughly.

Perhaps the best method of sampling the whole cane is to await its arrival at the factory, then grind several cart- or car-loads apart from other cane, and analyze the juice.

**267. Sampling Bagasse.**—To a certain extent the bagasse presents the same difficulties as the whole cane. The bagasse, however, is more or less well mixed in its passage through the mills and irregularities may be overcome by sampling across the conveyor.

Samples of bagasse should include all of that on a section of the bagasse-carrier its entire width and should be transported to the laboratory in clean galvanized iron cans, tightly covered. It should be remembered that loss of moisture in the sample introduces a very sensible error in the estimation of the moisture content. The sampling, sub-sampling, comminuting and handling of bagasse samples should be done as rapidly as possible with a minimum of exposure to the air. On reaching the laboratory the sample should be quickly and thoroughly mixed and sub-sampled. The small sample may be analyzed immediately or stored six hours in a closed box in the presence of chloroform and ammonia. A sponge should be saturated with the preservatives in the proportion of one part chloroform to six parts of strong ammonia and be attached to the inside of the box-cover.<sup>1</sup>

**268. Sampling the Juice.**—When saturation or imbibition is practiced it is necessary to secure two samples of the mill-juice, one from the first mill and the second from all the mills combined. It is preferable to draw the first of these samples from the juice canal of the crusher when there is liability of water from the mill-bearings mixing with the juice and in any event, it is usually advisable to sample at this point. The degree Brix of this first sample is used as a basis in calculating the so-called normal juice or undiluted juice, from the analysis of the mixed juices from the train of mills. The degrees Brix of the two samples are also used in calculating the dilution of the mixed juice due to maceration or leakage of water from the mill-journals.

**269. Dry Milling Factor.**—Special sampling is necessary in determining the factor or coefficient to be applied in reducing the degree Brix of the crusher or first mill and crusher-juice, to terms of the normal juice that would be yielded in milling of equal efficiency without maceration water. This factor should

<sup>1</sup> Spencer. Ind. Eng. Chem., Vol. 12 (1920), p. 1197.

be determined a sufficient number of times in the season to note its variations with cane and milling conditions. The juice from the crusher (or crusher and first mill) and that from the entire system are separately sampled during the period when the mills are operating with no maceration water whatsoever. The sampling should be by automatic devices and should be continuous for an hour or longer. The first sample is of relatively high degree Brix, sucrose content and purity, due to the comparatively moderate pressure applied in crushing the cane. The degree Brix of the second sample is lower than that of the first on account of the very heavy crushing by the mills which extracts the rind-juice and many of the impurities of the cane. The factor is the relation between the degrees Brix of the two samples. The following example illustrates the calculation and use of the factor: Brix of the

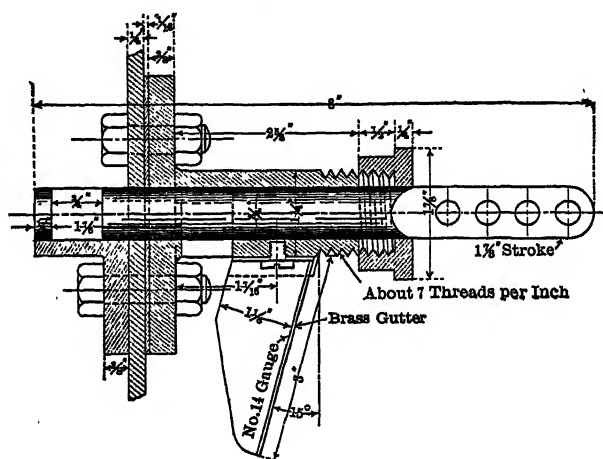


FIG. 107.—Calumet Juice Sampler.

two samples, respectively,  $20^{\circ}$  and  $19.7^{\circ}$ ; factor =  $19.7 \div 20 = 0.985$ . Brix of the mixed-juices in regular milling, 19.6, then  $19.6 \times 0.985 = 19.3$ , the degree Brix of the normal or undiluted juice.

**270. Sampling Devices.**—The juice should be sampled automatically and in proportion to that extracted. The milling is usually very uniform under good conditions of equipment and operation, hence samplers may be operated by some part of the mill mechanism, preferably a roll-shaft. Certain types of samplers may be driven by a reciprocating or other part of the juice-pump.

The Calumet is an efficient type of sampler (Fig. 107). It was devised at the Calumet Plantation, Louisiana. The Calumet sampler shown in the figure was designed by Dr. Spencer to draw juice from the crusher canal. A modified type is necessary for sampling juice under pressure in pipe-lines.

The construction of the Calumet sampler is shown in the sectional scale-drawing, with the device bolted to the crusher-canal. A hole is bored through

the plunger, near the end, to receive the sample. A lip or projection closes the lower side of the hole while filling. The plunger clears the trash from this projection at each stroke it makes. When the plunger is withdrawn it discharges the sample through the lower end of the hole into the gutter. A gland is provided to prevent leakage of juice.

The Calumet sampler for drawing juice from a pipe-line is of somewhat different construction. The plunger should be about 1 inch in diameter and have a stroke of about 3 inches. A juice cavity about  $\frac{1}{2}$  inch in diameter is drilled longitudinally into the end of the plunger and is fitted with a screw for adjusting the size of the cavity and consequently of the sample. A  $\frac{3}{8}$ -inch hole is drilled at right angles to the cavity and through it. Corresponding holes are drilled through the pump barrel for the inlet of air and the discharge of the sample from the plunger. Two rings of packing, controlled by a follower and packing-ring, are placed around the plunger, one at the outer end of the barrel and the other between the juice-inlet and outlet. There should be an oval opening in the packing-ring where it passes the juice-outlet, to admit of adjustment. The barrel of the sampler is screwed into the pipe from which the juice is to be drawn and is clamped into place with a locknut. This sampler may be operated from a reciprocating part of the juice-pump, reducing the speed, if need be, by a mechanism such as used in pumping oil to a bearing. The vertical- or outlet-hole in the plunger is made small to reduce the cutting of the packing.

A more recent sampling device known as the Mercedita sampler specially designed for taking samples of crusher juices is shown in Fig. 108 and consists

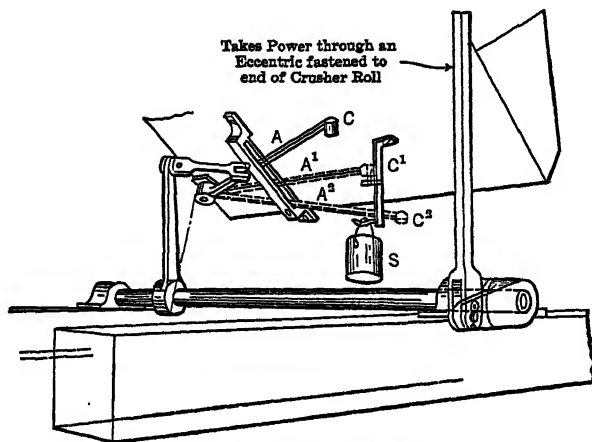


FIG. 108.—Mercedita Juice Sampler.

essentially of a link motion operated from a crusher-roll which actuates a swinging arm A carrying a small sample cup C. At each rotation of the roll

the arm and cup are swung out into the falling juice (position  $A^2 - C^2$ ) and then withdrawn, the cup being turned upside-down ( $A^1 - C^2$ ) by a tilting device as it comes over the sample containers. This arrangement has been found superior to any other sampler in simplicity and in freedom from clogging and fouling.

A device that is sometimes advocated is a heavy wire leading from the stream of juice to the sample bottle. With a short wire the error from evaporation is slight with cold juices, but with hot juices the evaporation is sufficient to affect the figures appreciably. The method should not be used where other means of sampling are available.

An undershot water-wheel, just dipping into the juice and driven by the current, may be used to sample from a canal. The axle of the wheel should be hollow and a few of the spokes should be tubular and communicate through the axle with the sample jar. The hollow spokes should terminate in small spoons which serve to take up a little of the juice and deliver it through the spokes and axle to the jar.

The difficulty of keeping small diameter pipes clean is an argument against arrangements such as these.

A can with conical cover, in which is located a small hole covered with gauze, is sometimes used. This is placed where the juice is falling and the stream flowing over the gauze keeps it clear of fiber, while a small amount of juice enters the can.

Samples may be drawn from the discharge-pipe of a direct-acting pump by means of a spring-controlled relief-valve. The valve should be adjusted to open at the moment of highest pressure at each pump stroke.

The difficulty with most of these samplers, except the Calumet and Mercedita types, is their liability to clog, tendency to foul, uncertainty as to their sampling in proportion to the quantity of liquid passing them and the probability of not drawing an average sample under certain conditions, *e.g.*, when drawing from a canal into which the juices from several mills flow. The Calumet sampler necessarily draws a correct sample when it is connected with the discharge line from a pump and is operated by the latter. When this sampler is connected with the canal leading from the crusher its sample is from only a part of the juice, but it is usually a very good approximation of a representative sample.

Particles of juice-soaked bagasse have been found to hang up on the projecting parts of the Calumet with consequent souring and for this reason the Mercedita sampler is preferred. The value of a mechanical sampler is in direct ratio with the ability to keep it clean. Hand sampling, where the honesty and dependability of the sampler can be checked up regularly, is second only to the best mechanical devices, while drip samplers are not generally to be recommended.

**271. Sampling the Mixed Juice.**—The accuracy of the factory control is based on the analysis of the mixed juices so the samples of this material should be taken with extreme care. Where weighing tanks are used (as in all modern plants) the best method is to have the scale-man take a measure of juice from each weighing-tank as it is being discharged. These small samples are poured



into a wide-mouthed container which is taken to the laboratory at the end of each hour. The container should be replaced by a clean one for the succeeding hour's samples. The sample containers should be well stoppered between subsamples. Because of the importance of this mixed juice sample it is considered advisable to keep the sample at the juice tanks cool by surrounding the container with ice. This obviates any possibility of deterioration during this hour when the sample is kept without any chemical preservative.

Automatic sampling of the mixed juices is complicated, particularly in a factory operating more than one tandem of mills, and does not give an accurately proportionated sample. Drawing a small amount from each weighing tank as described and compositing the hourly samples in the laboratory according to the number of weighing tanks filled is the only exact way of assuring a weighted sample.

Duplicate samples should be drawn, one for the density determination and the other for the polarization.

Methods for the preservation of the samples of juice and observations in regard to them are given in Sec. 273.

**272. Care of Samplers and Containers.**—In order that the sampler itself may not be a source of infection and cause the decomposition of the samples, it must be kept thoroughly clean and be frequently sterilized. Cleansing by means of a steam-jet is usually the most convenient and efficient method. All sampling devices should be thoroughly sterilized several times daily. The sample jars should be washed with hot water after each use and be thoroughly dried. The chemist should fully realize that in analyzing samples that are improperly drawn or cared for he is wasting his time and is obtaining misleading results.

Where measured samples are drawn at intervals by the workman, they may be conveniently stored in wide-mouthed, glass-stoppered jars. The stopper should have a small perforation in it to prevent sticking when the temperature of the factory falls. A convenient size for the jars is three liters and the mouth should be fully 13 cm. (5 inches) in diameter. The object of the large mouth is to obviate the use of a funnel and to prevent the workmen from spilling juice on the edges of the jar. Small metal-cups, with long handles, are convenient for measuring the samples. The size of the cups depends upon the number of tanks that are filled daily; usually a 3-5 cc. cup for the sucrose sample and 10-15 cc. for the density sample are suitable sizes. These cups should be thoroughly rinsed with juice each time before drawing the samples, and after the addition of samples the contents of the jars should be thoroughly mixed.

**273. Preservation of Samples.**—A preservative must be added to the samples in compositing. Samples for use in the Brix and ash determinations may be preserved by the addition of mercuric chloride in the proportion of 1 part to 5000 parts of juice. One-half a milliliter of a saturated alcoholic solution of the salt per liter of juice will give this proportion. These samples may also be preserved with formalin (40 per cent formaldehyde) using 0.3 to 0.5 ml. per liter, but the mercuric chloride is preferable. Formaldehyde should never be used in compositing samples for glucose determinations as it is copper reducing. Dr. Spencer discontinued the use of formaldehyde as a preservative

for laboratory samples about 1919.<sup>2</sup> Its use in storing juices in the factory during enforced shut-downs is valuable, although it is recognized that some inversion due to the acidity of the juice takes place in its presence (p. 85).

The most satisfactory preservative of juices for sucrose and glucose tests is Horne's dry subacetate of lead. This is very efficient when used in the proportions of 20 grams per liter of juice. Spencer made many tests in Cuba with this salt and found that this proportion would keep juices without change in polarization for several days. The curves in Fig. 109 show the rates of deterioration with various amounts of dry lead in one set of these tests.<sup>3</sup> The lead should be weighed and added to the sample in proportion to the amount of juice added to the composite. For example, if 200 ml. are to be composited each hour, 2 grams of the dry lead salt should be added with this portion of juice, the whole being thoroughly shaken. The practice of adding

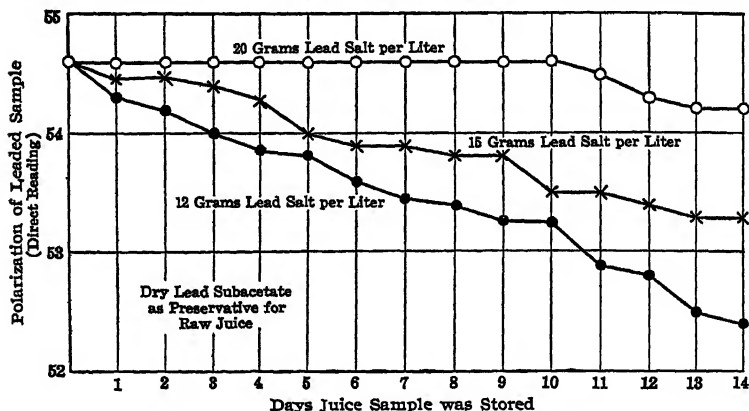


FIG. 109.—Preservation of Juice with Horne's Dry Lead.

all the preservative required for the completed sample to the container before compositing is started should never be permitted.

If the subacetate of lead is used in solution, an estimate of the probable volume of juice that will be included in the day's composite sample should be made and for each estimated 100 cc. 5 cc. of the lead solution should be accurately measured into the sample-jar. At the close of the day's work the volume of the sample and preservative should be ascertained and then sufficient water should be added to make the total dilution 10 per cent of the volume of the juice itself. This enables the use of Schmitz's table for sucrose.

It is preferable to composite the samples in the laboratory itself, thus giving

<sup>2</sup> Ind. Eng. Chem. Vol. 12 (1920), p. 1197.

<sup>3</sup> Recent work by E. Lumeau (Sugar, Nov., 1928) indicates that 6 grams of dry lead per liter are sufficient for juice storage. Dr. Spencer's investigations showed juices to be highly variable as to the amount of the preservative required; 20 grams per liter being the safe limit for all juices. In his earlier work, he prescribed 12 grams, later raising this to 15, and finally to 20 grams per liter.

the chemist a good control over the sample-boys, and making sure that the subsamples are drawn at regular intervals.

**274. Sampling the Filter-press Cake.**—The composition of the filter-press cake varies in different parts of the press and of the cake itself. This makes strictly accurate sampling impracticable since this work must be left in the hands of the pressmen. The best approximations and comparisons are obtained by cutting pieces of the cake systematically from various parts of the press.

The instrument shown in Fig. 110 is very suitable for this sampling. It is made of heavy brass and of such a size that it may readily be grasped by the hand over the cover. This latter is fastened to the body or receptacle by bayonet catches and a set-screw. The cutter is a brass-tube  $\frac{1}{4}$  inch thick and about  $\frac{1}{2}$  inch in diameter at the cutting edge. The body of the tube is coned towards the receptacle, so that the plug of cake will readily pass into the latter. The cutting edge should be of the thickness of the tube to prevent damage to the filter cloth or a guard should be placed on the cutter to prevent complete penetration of the cake.

Several of these samplers should be provided so that one may be filled from each press. The pressmen should cut a number of plugs in the usual procedure, *e.g.*, from various parts of the second cake, then from the fifth cake and so on. The plugs are accumulated in the receptacle, one remaining in the cutter and closing it. A small tube, open at both ends, is attached to the inside of the cover and holds a sponge saturated with formaldehyde, for the preservation of the sample.

A mixture of one part chloroform and six parts strong ammonia is preferable to the formaldehyde. The sample should be sent to the laboratory each time a press is filled. This provides a check on the sampling and count of presses cleaned. The sampler should be thoroughly cleaned after removal of the plugs and returned to the presses.

The sample may also be obtained by means of a brass-tube, fitted with a piston for removing the plugs. A cork-borer may be employed for the purpose,

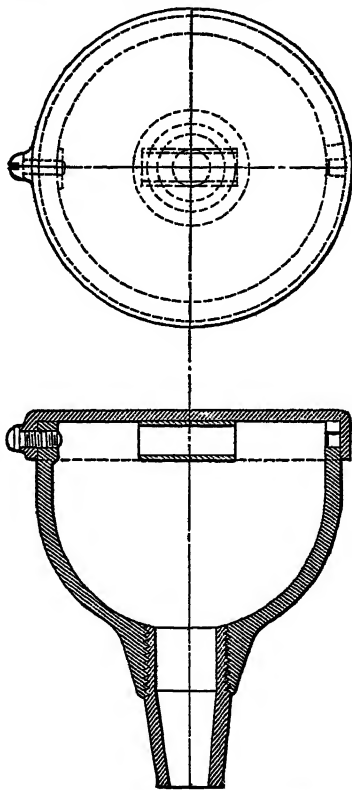


FIG. 110.—Sampler for Filter-Press Cake.

but must be used with care on account of risk of cutting the filter cloth. The plugs should be stored in a covered vessel in an atmosphere saturated with ammonia and chloroform.

It is difficult to control or check the sampling of the press-cake, especially as there is a natural tendency for the pressmen to sample only the hardest parts of the cakes. With the use of systematic methods of washing the cake the error of sampling, however, becomes less important as the loss of sugar is small.

The samples, collected as has been described, are not usually separately analyzed, but are composited, preferably during six-hour periods. Each time that a filled receptacle is received by the laboratory, the plugs of press-cake should be removed and chopped into fine pieces and be thoroughly mixed. A measured portion of the minced cake from each press should be placed in a jar in an atmosphere of the chloroform-ammonia mixture as above. The united subsamples should be thoroughly mixed and analyzed once every six hours.

If preferred, the subsamples may be composited during a twenty-four-hour period as follows: Weigh 25 grams of the six-hours' sample and wash it into a glass mortar with water and 6 ml. of lead solution (437). Rub the cake to a paste to thoroughly incorporate the lead-salt with it. Cover the mortar pending the receipt of the next sample and proceed as before, adding the second portion to the first, with additional lead. The third and fourth subsamples are treated in the same way and finally the mixture is washed into a 400 ml. flask, diluted to the mark and polarized (321).

**275. Sampling Sirups.**—In cases where the sirup may not be thoroughly mixed in the tank before sampling, a "thief" sampler may be used. This consists of a tube, long enough to reach the bottom of the tank and provided with a suitable ball or other type of valve. The tube is passed through the sirup, to the bottom of the tank, and on its removal takes with it a sample of each layer of sirup.

In factories which pump all of the sirup through a single-pipe line to the storage-tanks, this material may be sampled by a Calumet pipe-line type of sampler (270) and with increased accuracy as compared with hand methods. This method is especially desirable when the sirup analysis is used as a basis for the calculation of available sugar. Probably as satisfactory a method as any is to have the multiple effect operator take a small portion of the sirup every fifteen minutes as it leaves the effect, compositing it without preservative.

**276. Sampling Massecuite and Molasses.**—The grain-massecuites are not usually of uniform density or composition. This lack of uniformity is caused by imperfect circulation in the vacuum-pan. This is especially noticeable in very dry massecuite boiled without the addition of molasses. The sample of massecuite should therefore be drawn, little by little, as the strike is being discharged from the pan if the greatest accuracy is required. In ordinary practice a "grab" sample taken as the pan is discharging will serve, or if preferred, portions may be taken at the beginning, the middle, and the end of the strike as it runs from the pan.

If the samples of the several strikes of massecuite included in the day's work are to be composited to form a single sample, a measured portion of each

must be used. Sugar-boilers usually fill the pans to the same point; in such cases, if the pans are of uniform size, one measure will answer for all, otherwise there should be a set of proportionate measures, one for each pan. A small stemless funnel of glass forms a convenient measure.

Molasses is sampled in the same way as sirup, and, according to the exigencies of the work, the samples are analyzed separately or are composited. A composite sample of the final molasses should be prepared from time to time for the determination of the sucrose by the Clerget method and the solids by drying for the calculation of the true purity. These samples should represent a definite quantity of the material.

**277. Sampling Sugar.**—The workman at the sugar-scale should remove a sample from each package of sugar as he weighs it. The samples should be thrown into a tin box provided with a funnel-shaped opening to receive them, as shown in Fig. 111. The can is conveniently placed on a shelf at the scaleman's side in order to prevent stooping. The composite samples so obtained should be analyzed at frequent intervals, preferably an analysis should be made every six hours,

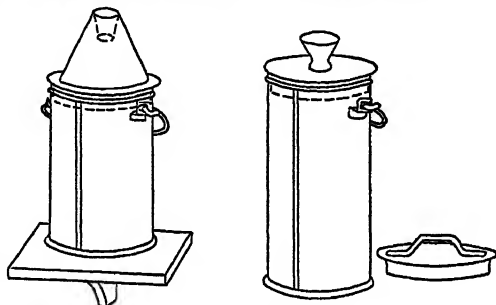


FIG. 111.—Sample Cans for Sugars.

and the number of packages represented by the sample should be recorded for use in calculating the averages. The can shown at the right of the figure is the type generally used in refineries where raw sugar is received in packages. The funnel-top of the can is replaced by a closed top when the can is full or the lot of bags complete.

In sampling sugar in the packages a "trier" is usually employed (Fig. 112). The trier is a long trough-like instrument, which, being plunged into a quantity



FIG. 112.—Trier for Sampling Sugars.

of sugar, will, on withdrawal, remove a sample representative of the sugar through which it has passed. This sample should be thoroughly mixed, reducing all lumps, and exposing it to the air as short a time as possible.

The following are the U. S. Treasury instructions for sampling sugars:

Sugar in hogsheads and other wooden packages shall be sampled by putting the long trier diagonally through the package from chime to chime, one trierful

to constitute a sample, except in cases of small marks, when an equal number of trierfuls shall be taken from each package of the mark to furnish the required amount of sugar necessary to carry out the provisions of the regulations. In the sampling of baskets, bags, ceroons, and mats the short trier will be used, care being exercised to take the sample fairly from the central contents of the packages, and in such manner that the samples from each class of packages shall be uniform in quantity. When the hard condition of the sugar renders the use of the short triers impracticable, the knife may be used.

The Treasury regulations require the sampling of all the packages in a lot, instead of a certain proportion of them, as formerly. This conforms to commercial usage.

## CHAPTER XXV

### ANALYSIS OF THE SUGAR-CANE

**278. Estimation of the Sucrose—Direct Method.**—The first step in the analysis of the cane is the preparation of the sample. As has been shown a representative sample of the cane without special apparatus is almost impossible to obtain. With the usual facilities, such as a knife or shears, the sample of whole cane can neither be prepared rapidly enough to avoid error by evaporation, nor in a suitable state of division for a thorough extraction of the sucrose. In the analysis of whole canes it should be noted that the composition varies greatly in different parts of the stalks, thus complicating the preparation of the sample.

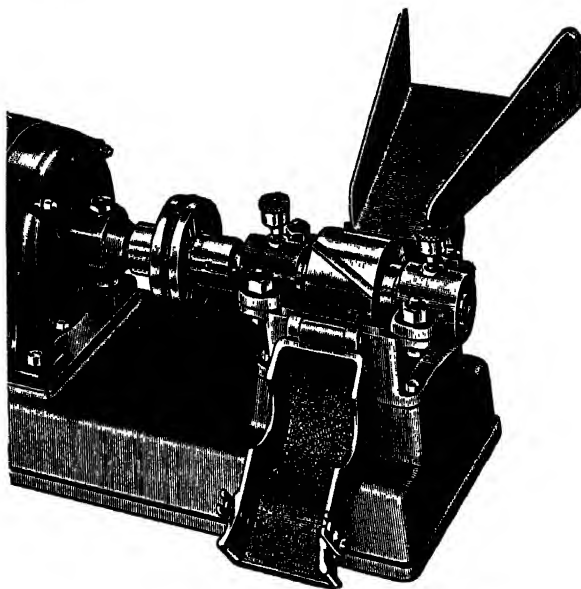


FIG. 113.—Warmouth-Hyatt Cane Disintegrator.

Whole cane can be rapidly and properly shredded by means of the Warmouth-Hyatt Disintegrator, Fig. 113. Knives are fitted into the revolving shaft which is connected directly to the motor. In use the cover, shown open in the illustration, is closed and bolted and the cane (cut into short pieces) is fed down the hopper and is shredded, dropping through an opening in the base, not shown. The machine is usually mounted on a table with a drawer under-

neath in which the sample is collected. The knives can be removed for sharpening or replacing.

For the direct estimation of the sucrose in the whole cane proceed as follows: Place 100 grams of cane prepared as above in a suitable dish or beaker and add approximately 200 ml. of boiling water and boil during ten minutes; carefully drain off the liquid and add another portion of 200 ml. of water, and again digest during ten minutes. Repeat these digestions with water in all seven times, and after the last press the residue in a hydraulic or other powerful press, uniting the portions of solution drained from the chips. Cool the liquid and weigh it and also determine its degree Brix. To approximately 100 ml. of this solution in a sugar-flask add dry subacetate of lead for clarification. After thorough mixing and filtration polarize the filtrate, using a 400-mm. observation-tube. Divide the polariscope reading by 2, since a double-length tube is used, and calculate the per cent sucrose in the solution by Schmitz's table, page 481. From the per cent sucrose in the solution and the weight of the latter, calculate the weight of sucrose. This number is the weight of sucrose in 100 grams of cane or the percentage of sucrose in the cane.

**279. Estimation of the Sucrose. Indirect Methods.**—In the earlier days of laboratory control of cane-sugar factories, the sucrose was estimated by applying the percentage of sucrose in the normal juice to its weight as derived by deducting the marc (fiber) from 100. O. H. Francis<sup>1</sup> called attention to the error of this method in assuming the juice of the cane to be homogeneous. Many other experimenters have also shown that the composition of the juice varies in the same part of the stalk and that the cane contains water, termed colloidal water by Scheibler, that is almost or quite free of sugar. If a piece of a stalk of cane be entered into a small hand-mill and be slowly pressed, water will drip from the free end. In view of these considerations it is evident that an indirect estimation of the sucrose content of the cane must include the use of a factor to reduce to terms of the undiluted juice. This factor is necessarily quite variable and no great reliance should be placed upon analyses in which it is used.

The above method is discussed here on account of its use by many chemists and its bearing upon methods of stating the efficiency of mill-work.

As in the beet-sugar industry, it is the custom of many chemists to deduce a factor from experimental data, corresponding to the percentage of normal juice in the cane, and apply this number in calculating the sucrose by the indirect method. In order that such a factor may be applicable, the experimental data must be obtained under the same milling conditions as in the extraction of the juice sample upon which to base the calculations. Such a factor can only be properly calculated from data obtained by actual experiment with the factory mills and even when so deduced is but an approximation, since the composition of the cane is constantly changing.

The following is the customary and probably the best method of indirect analysis of the cane in working with or without saturation. The weight of the cane is taken as reported by the cane department; the weight of mixed or

<sup>1</sup>Trans. Roy. Soc. Canada, 1894, 1, 101.  
<sup>2</sup>Trans. Roy. Soc. Canada, 1894, 1, 101.



diluted juices from all the mills is ascertained by direct weighing or by measurement and calculation by the laboratory; the weight of bagasse is estimated by deducting the weight of the diluted juice from the sum of the weights of the cane and the saturation-water; the sucrose is determined in the juice and the bagasse by direct analysis. Manifestly the weight of sucrose in the cane is the sum of the weights of sucrose in the juice and bagasse, and this number divided by the weight of the cane and multiplied by 100 gives the percentage of sucrose in the cane.

There are several conditions that may lead to inaccuracy in the above method. The juice may be diluted by leakage of the water used in cooling the mill-journals; the bagasse parts with more or less moisture by evaporation in passing through the mills; there may be inaccuracies of weights, measures and analyses. The first of these need not be expected where modern mills of good construction are operated. With the old types of mills, however, it is often necessary to run cooling water upon the bearings and a part of this is liable to leak into the juice. Such leakage may usually be detected by noting the relation between the percentage of saturation-water and the dilution number. The error from the evaporation of the moisture cannot be eliminated, but is probably usually very constant in a given milling-plant. Inaccuracies of weights, sampling and analysis are usually avoidable. Inaccuracy in the measurement or weight of the saturation-water, which is used in calculating the weight of the bagasse, should be avoidable, but is a frequent source of error.

The impossibility of accurately sampling whole canes usually precludes the use of the direct method of analysis in factory control, hence the method just described should be given preference.

**280. Determination of the Woody Fiber or Marc.**—The samples should be very finely shredded. Transfer 50 grams of the material to a tared beaker of 400 ml. capacity. Stretch a piece of washed linen over the top of the beaker, fastening it in place by a strong rubber band. An opening should be left in the linen, opposite the lip of the beaker for replenishing the water. The linen is designed to serve as a filter. Digest the cane-shreds two times of ten minutes each with warm distilled water, not hotter than 75° C., pouring off the solution each time through the linen, and washing back into the beaker the fragments of cane that adhere to the filter. After the digestions with warm water, digest the cane-shreds five times, of ten minutes each, in boiling water pouring off the liquid as before. Dry the beaker and residue in an oven at 100° C., until there is no further loss of weight, or until there is a slight gain over the previous weight. Use the smaller weight in the calculation. The weight of the residue multiplied by 2 is the percentage of fiber or marc in the cane.

The dried fiber and linen attract moisture with extreme rapidity; therefore the beaker and contents should be cooled in a desiccator and then weighed as quickly as possible. Preparatory to weighing the beaker and residue, their approximate weight should be placed on one of the balance-pans.

Special apparatus should be employed where frequent direct fiber-determinations are required. This should be capable of operating with compara-

tively large quantities of cane or bagasse in order to reduce the sampling error. A convenient apparatus is shown in Fig. 114. This is a simple form of Soxhlet's extractor and is made of thin copper or brass. The body of the extractor, *A*, is approximately 3.5 inches in diameter and 12 inches long. Lugs are provided to support a cylinder, *B*, about 3 inches above the bottom of the extractor. The cylinder has a removable cover of 80-mesh wire-gauze and the bottom is of the same material. A cylinder of this size will hold from 50 to 100 grams of shredded bagasse or a larger weight of cane. The chamber below the lugs is to provide drainage space. A weighed quantity of shredded cane or bagasse in the cylinder *B* is placed in the extractor and a slow stream of cold

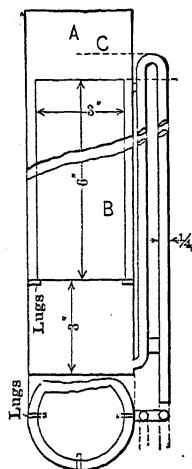


FIG. 114.—Extractor for Use in Fiber Tests.

water is run upon it. The water is quickly removed by the syphon-tube when it reaches the level *C*. The rate of syphoning may be controlled by partly closing the outlet with a plug of wood. The lower chamber should be large enough and the rate of syphoning slow enough to permit partial drainage of the cylinder before the water again rises to the level of the cane. Hot water should be substituted for the cold after a few extractions. If desired the extractor may be heated with a lamp, but in this event the lower chamber should be deepened and the outlet should be an inch or more above the bottom.

After thorough extraction, close the water-inlet and drain the fiber. Remove the cylinder from the extractor and place it in an oven to dry the fiber, or more properly speaking, the "marc." Drying may be promoted by soaking the marc in strong alcohol. A cylinder of the water-saturated marc may be dried in about four hours in the special bagasse-oven, Fig. 123. The cylinders and extractor should be made with a view to the use of an oven of this type, and therefore much larger than has been indicated.

According to Pellet,<sup>2</sup> the use of alcohol instead of water in marc determinations is objectionable. The alcohol precipitates silica, phosphates, and nitrogenous and non-nitrogenous matters, which are held by the fiber and exaggerate its quantity.

There are several processes of direct-fiber determination, using special apparatus, that are accurate as analytical methods for the particular sample, but they use such small portions per test that the sampling error becomes quite large, and the results valueless.

The following indirect methods of fiber determination are those usually employed in mill control the second method being preferable: (1) The bagasse is sampled and the fiber is directly determined in it. The bagasse produced per cent cane  $\times$  the per cent of fiber in the bagasse  $\times$  100 = per cent fiber in cane. (2) The per cent sucrose in the bagasse and the purity of the residual juice and the moisture are determined. The percentage of juice solids in the

<sup>2</sup> Bulletin Assoc. des Chimistes, 22 April, 1905.

bagasse is calculated from the percentage of sucrose and the purity of the residual juice. The sum of the juice solids and the moisture deducted from 100 give the percentage of fiber in the bagasse. The purity of the juice flowing from the bagasse-roll of the last mill in the train is considered to be that of the residual juice.

The following example illustrates the second indirect method: Per cent of bagasse, 25; sucrose in the bagasse, 4 per cent; moisture, 48 per cent; residual juice purity, 78 per cent; then  $4 \div .78 = 5.13$  per cent juice-solids;  $100 - (5.13 + 48) = 46.87$  per cent, fiber in the bagasse;  $25 \times .4687 = 11.72$  per cent, fiber or marc in the cane.

Dr. Spencer compared a large number of analyses by method (1) with the calculated fiber by method (2). The agreement by the two methods was so good that he discontinued the direct determinations of fiber in the bagasse in the factories under his supervision. (*See* also 325.)

## CHAPTER XXVI

### ANALYSIS OF THE JUICE

**281. Determination of the Density.**—The density of the juice is almost always determined by means of an hydrometer (243) though a pyknometer (245) or the Westphal balance (244) may be used. The Brix spindle is the more convenient hydrometer for this purpose, since its readings are used in calculating the coefficient of purity.

The readings on the Baumé scale, or the specific gravity as determined by the Westphal balance or pyknometer, may be converted into degrees on the Brix scale by means of the table on page 451 or page 459, according to the standard temperature selected.

The juice is first thoroughly strained through a fine-mesh screen (a cone of wire gauze is convenient) to remove particles of bagasse fiber and other suspended matter. Samples of juice for Brixing or drying should be preserved with either mercuric chloride or formaldehyde, preferably the former. Lead preserved juices should never be used for these determinations.

In using the hydrometer, fill a wide cylinder to the brim with the sample of juice and set it aside for the escape of air-bubbles. The time required for this varies from a few minutes to half an hour, but usually ten minutes suffice. A vacuum connection as described on page 255 facilitates the removal of the air bubbles and should be used in all routine work.

The spindle should be lowered into the cylinder, after the escape of the bubbles, causing the juice to overflow and carry away with it the froth and mechanical impurities floating upon the surface. It is well to blow on the surface of the juice as it overflows to help remove the froth. The spindle should now be lowered farther into the juice, until it floats, care being taken to see that the stem is wet for a few tenths above the point of which the spindle will come to rest.

After allowing sufficient time for the temperature of the spindle to reach that of the juice, read the scale as directed in 243 and illustrated in Fig. 89. The temperature of the juice should be noted for use in correcting the observed density.

The temperature correction when using spindles whose normal is  $17\frac{1}{2}^{\circ}$  C. is made with the aid of the table, page 458. For example: Let  $18.15^{\circ}$  Brix at  $24^{\circ}$  C. be the observed density and temperature. Referring to the table of corrections, under the heading "Approximate degree Brix and Correction," follow down the column 20, the degree Brix nearest  $18^{\circ}$ , to opposite the temperature  $24^{\circ}$  C., and take off the correction .44, which must be added to 18.15, making the corrected degree Brix, 18.59. Had the temperature been below

17½° C., the correction would have been subtractive. Similarly, the table on page 469 is used in making temperature corrections for instruments standard at 20°/4° C. The table of comparisons for these instruments is given in page 459.

**282. True Degree Brix or Total Solids by Drying.**—The juice sample should be weighed by difference from a weighing bottle, preferably one fitted with a ground-in pipette and rubber bulb. Use one of the methods of drying on pumice or quartz sand (Sec. 247) or the Spencer oven, Type "A," absorbing the juice on asbestos (Sec. 248). In any case employ sufficient juice to give about 1 gram of dry matter. Observe precautions for protecting the sample from absorbing moisture after drying (Sec. 246).

The weight of dry matter divided by the weight of juice used, multiplied by 100 = per cent of total solids.

**283. Total Solids from the Refractive Index.**—The refractometer may be used for estimating the solids as described in 249. The dipping refractometer may also be used for juices.

**284. Determination of the Sucrose.<sup>1</sup> Special Pipette for Measurements.**—This method is generally used only for juices that have not been preserved with subacetate of lead. The pipette,<sup>2</sup> Fig. 115, is so graduated that if filled to the mark corresponding with the observed (uncorrected) degree Brix, it will deliver two normal weights of the liquid. Makers now graduate these pipettes to deliver either 52.096 grams for use with the older system of Mohr's 100 cc. flasks at 17½° C. or 52 grams with the 20° C. standard and 100 ml. flasks. The usual graduation is for a range of densities from 5° to 25° Brix in tenths. These instruments, called sucrose pipettes, are preferably made with a delivery tube about 4 inches long. With the short tube the pipette may be supported by the flask while draining, leaving the chemist free to continue a series of such measurements with other pipettes.

In using this pipette in the analysis of a juice, proceed as follows: Determine the density of the juice with a Brix hydrometer, noting the degree Brix without temperature correction. Fill the pipette with juice to the mark corresponding with its observed degree Brix, and discharge it into a 100-ml. flask. Add 3 to 5 ml. of diluted lead subacetate solution (437), complete the volume to 100 ml. with water, mix thoroughly and filter the contents of the flask. Polarize the filtrate, using a 200-mm. tube, and divide the polariscope reading by 2 to obtain the percentage of sucrose.

<sup>1</sup> The use of "sucrose" as synonymous with "polarization" is general in the sugar industry and for convenience the practice is continued in this book. Results with double polarization methods are termed "True Sucrose," "Clarget Sucrose," or simply the "Clarget."

<sup>2</sup> This pipette was devised by C. A. Crampton and G. L. Spencer, independently and at about the same time. It is termed "Crampton's" or "Spencer's sucrose pipette" by the dealers.

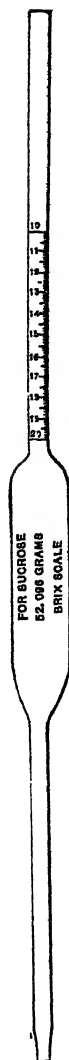


FIG. 115.  
Sucrose  
Pipette.

Factor for 78.5.....	132.25
Correction for 25.7° C.....	3.02

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129.23

78.5/129.23 = 60.75

Consulting Schmitz's Table as before:

Opposite 60 in column for 18° Brix.....	14.55
Interpolating for 0.75.....	.18

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Clerget Sucrose..... 14.73

The leaded composite sample for Clerget work may be kept as long as five days if proper precautions as to sterilizing containers and protecting the sample from contamination are observed.

**287. Glucose (Reducing Sugars).—Preparation of the Sample.**—If the juice sample contains no preservative it may be filtered directly through dry kieselguhr, no lead or other clarification being used. (Sec. 230.) Samples preserved with mercuric chloride may also be treated this same way, but juices containing formaldehyde should never be used for glucose determinations as formaldehyde reduces copper. Before adding the kieselguhr it may be well to add dry sodium oxalate (about .25 gram for each 100 ml. of juice) to remove lime salts. (See page 239.)

For juice samples preserved twenty-four hours with Horne's dry lead (20 grams per liter of juice) Harris <sup>6</sup> has shown that results corresponding with the glucose found in the fresh juice untreated (filtration through kieselguhr only) may be obtained by the following procedure: To the well-mixed leaded juice, unfiltered, add 0.75 gram of powdered oxalic acid for each 100 ml. of sample, shake thoroughly, allow to stand a few minutes, and then filter. By means of a Spencer glucose pipette (use of which will be explained below) make up a solution of this delead filtrate of such strength that 50 ml. will contain 5, 10, 15, 20 or 30 grams of the juice, depending on the amount of glucose present. Taking 50 ml. of this filtrate, proceed with the Herzfeld method of gravimetric glucose (233), determining the copper as cupric oxide or as copper by the Vorticek-Wedderburn method, 234 or as both, using one method as a check on the other. From the weight of copper the glucose may be found by reference to the table on page 517, according to the percentage of sucrose in the juice.

In his investigation, Harris showed that neutral salts (potassium or sodium oxalate, sodium phosphate, and others) are useless as reagents to break up the lead levulosate formed by the presence of the basic lead salt. He also proved that results are valueless if the lead precipitate is filtered off and the deleading agent added to the filtrate. This method, using oxalic acid to delead lead-preserved juices for glucose work has proved of real value for control tests and the establishment of the "glucose balance" of the factory.

The Spencer glucose pipette is similar in principal to the sucrose pipette described in Sec. 284 in that it delivers a certain weight of juice corresponding

to the observed Brix (*i.e.*, uncorrected for temperature). In the case of lead-preserved juices the Brix is taken on a parallel sample preserved with mercuric chloride or formaldehyde as previously described. The pipettes listed in the instrument-maker's catalogues are generally made to deliver 50 grams, in which case, by adding the 50 grams so delivered to a 500 ml. flask and making to the mark with water, each 50 ml. will contain 5 grams of juice, while the use of a 250 ml. flask will give 10 grams per 50 ml.; by delivering two volumes of juice from the pipette, 20 grams per 50 ml.; a second pipette calibrated to deliver 60 grams may be used with a 200 ml. flask to give 15 grams, and two volumes to 200 ml. will give 30 grams per 50 ml. Many laboratories carry glucose pipettes calibrated for 20, 30, 40 and 80 grams using a 200 ml. flask to obtain the dilution desired.

McAllep and Cook <sup>7</sup> recommend neutral lead acetate clarification for juices for control tests (presumably with samples preserved with mercuric chloride), then filtering and adding disodium phosphate—potassium oxalate solution (see page 239) to an aliquot portion of the filtrate to remove all lime and lead. The amount of lead solution specified is 1 gram per 10 grams of juice with 3 ml. of the phosphate-oxalate mixture. The Eynon-Lane volumetric method (Sec. 239) is recommended by them, the solution of juice being so prepared that from 25 to 40 ml. (125 to 200 mgs. of glucose) will be used in the titration. It is evident that this volumetric method may be used with lead-preserved juices, delead according to the directions given by Harris <sup>8</sup> with powdered oxalic acid.

**288. Determination of the Ash.**—*Carbonated Ash or Normal Ash.*—The carbonated ash is usually determined only in research work and not in commercial analysis. Dry 10 grams of juice in a weighed platinum dish, and proceed to incinerate the residue as given in Sec. 252.

*Sulfated Ash.*—Dry 10 grams of the juice in a shallow tared fused silica or platinum dish. Proceed as in 251. Most sugar laboratories continue to deduct one-tenth from the weight of the sulfated ash to allow for the increase in the weight of ash due to the formation of sulfates instead of carbonates, although the official methods of the A. O. A. C. specify that the weight as found shall be taken as sulfated ash. Where the practice is to deduct one-tenth the calculation becomes: Weight of sulphated ash  $\times 9$  = per cent ash. If the A. O. A. C. method is followed: Weight of sulfated ash  $\times 10$  = per cent sulfated ash.

It is usually more convenient to measure 10 ml. of the juice for the ash determination rather than to weigh 10 grams. In such cases the calculation is modified by dividing by the specific gravity of the juice.

**289. Acidity of the Juice.**—*By Titration with Phenolphthalein.*—Normal cane-juice is always acid. The acidity is generally expressed in terms of the number of cubic centimeters (or millimeters) of deci-normal alkali (usually NaOH) required to neutralize 10 ml. of the juice, phenolphthalein being the indicator most commonly employed. It is necessary to state what indicator is used since the end point of different indicators is not the same. Neutrality

<sup>7</sup> Facts About Sugar. Vol. 23 (1928), p. 806.

<sup>8</sup> *Loc. cit.*

to phenolphthalein, for example, is definitely alkaline to litmus, to cite the two commonest indicators in sugar work.

To carry out the titration, pipette 10 ml. of the juice into a porcelain dish or casserole, dilute with about 25 ml. of water which is neutral to phenolphthalein, add two to three drops of neutralized phenolphthalein solution (Sec. 430) and then run into the mixture, from a burette, tenth normal sodium hydroxide solution (Sec. 448) until there is evidence of a pink color. Record the number of milliliters of the alkali used as the acidity of the juice. With dark colored juices it is sometimes difficult to distinguish the change in color promptly. As an aid to the eye place alongside the dish in which the titration is to be made a duplicate one containing the same juice and water mixture. If this is nearly neutralized with the caustic solution it will then be possible, by comparison with this blank, to note the first approach of the pink color as the alkali is added to the test sample.

*pH of the Juice.*—This is the most exact and valuable method of recording the acidity or alkalinity of juices. For a discussion of the theory and usefulness of the pH numbers, consult the chapter beginning p. 274. It must be clearly understood that acidity of the juice by titration and pH will bear no constant relation since the former is total acidity, while the H-ion concentration (pH) is the *effective* acidity.

The pH determination may be made by the spot method (page 280) or preferably by dilution with neutral water and comparison with standards in tubes as described in 258. The dilution for routine work should be fixed, say one part juice to five water. The pH is not generally made on the untreated raw juice but on the mixed juice, after the addition of lime, to determine the correct reaction for clarification (p. 46) and for this purpose bromthymol blue (6.0–7.6 pH) and cresol red (7.2–8.6) will cover all the desired ranges. The method outlined on p. 280, using diluted dye for the dilution of the juice simplifies the procedure, particularly if workmen are called upon to make the test.

#### 290. Phosphoric Acid Determination.—*Uranium Method.*—

- A. Dissolve 35 grams of chemically pure uranium acetate in distilled water. To this add 50 ml. glacial acetic acid and make up to 1 liter.
- B. Dissolve 100 grams of chemically pure sodium acetate in distilled water and add 50 ml. glacial acetic acid. Make up to 1 liter.

Add 1 ml.  $\text{NH}_4\text{OH}$  to 100 ml. of juice, acidify with acetic acid and add 10 ml. of solution B. Titrate with solution A, using powdered potassium ferrocyanide on a drop reaction plate as an indicator. The solution usually settles sufficiently to allow a small portion of clear liquor to be removed for the end point determination. When the end point has been reached the liquor gives a brown precipitate of uranium ferrocyanide when brought into contact with potassium ferrocyanide. Solution A should be standardized against an accurately weighed sample of chemically pure tricalcium phosphate, which ~~is~~ dissolved in nitric acid, precipitated by adding a slight excess of ~~ammonia~~ and redissolved in a moderate excess of acetic acid.



The greater portion of the uranium solution should be added in the cold but the titration should be finished at 90–100° C. The amount of  $P_2O_5$  present is calculated from the milliliters of uranium solution used.

*Phosphomolybdate Method.*—

A. Standard nitric acid.

B. Standard sodium or potassium hydroxide free from carbonate.

The acid solution must be compared to the alkali solution using phenolphthalein as an indicator. Solutions of convenient strength will be 0.0324 normal, one ml. of such a solution being equivalent to 0.1 mg. of  $P_2O_5$ . The caustic alkali solution should be freed from carbonate by treatment with barium hydroxide.

C. Ammonium molybdate solution. Dissolve 75 grams of ammonium molybdate in 500 ml. of water with the addition of a little ammonia. Filter if turbid and pour with constant stirring into 500 ml. of a mixture of 250 ml. of concentrated nitric acid (sp. gr. 1.40) and 250 ml. of water. Allow to stand for several days and filter off as used.

Ash 100 ml. of juice, dissolve the residue in nitric acid in a 250 ml. Erlenmeyer flask and make up to a convenient volume. If sulphuric acid is present remove by the addition of a few drops of  $BaCl_2$  solution. Nearly neutralize the acid solution with ammonia, warm to about 40° and add 10 ml. of the molybdate solution C. Digest the solution for ten or fifteen minutes, increasing the temperature from 40° C. by warming on the water bath. Close the flask with a rubber stopper and shake for five minutes.

Filter through a small filter paper and wash with nitric acid (15 ml. of concentrated acid to 1 liter of water) until the washings are free from molybdenum as shown by the absence of a precipitate with ammonium sulphide. Now wash with a potassium nitrate solution (1 gram in 1 liter of water) until all the nitric acid is removed.

Place the filter paper and phosphomolybdate precipitate in a beaker and add 10 ml. portions of the standard alkali until the precipitate is completely dissolved. Add a few drops of phenolphthalein and titrate the excess alkali with the standard acid. The amount of  $P_2O_5$  is calculated from the number of milliliters of alkali used.

*Pyrophosphate Method.*—Bond<sup>9</sup> in his work on defecation of sugar liquor determined  $P_2O_5$  by precipitating directly from the juice (after removal of  $CaO$ ) as magnesium ammonium phosphate, igniting to the pyrophosphate and weighing.

**291. Analysis of Clarified Juice.**—The analysis of the clarified juice is made by the same methods as that of the normal juice. If the carbonation process is used, which is the case in very few factories, the juice must receive an additional treatment with carbonic acid, after the first carbonation, and before the analysis, to precipitate all the lime it contains.

<sup>9</sup> J. D. Bond. *The Planter*, Vol. LXXIV (1925), No. 21.

## CHAPTER XXVII

### ANALYSIS OF THE SIRUP, MASSECUTES, AND MOLASSES

#### SIRUP

**292. Analysis of the Sirup.**—The tests usually required in the examination of a sirup are the density and the percentage of sucrose. In special investigations or in tracing inversion, glucose determinations are necessary and the sucrose should then be determined by Clerget's method, page 327. pH determinations are now usually made at intervals of from one to three hours as a routine procedure. The colorimetric method, diluting 1 : 3 or 1 : 5, as directed for the juice (289) should be used for this test.

The normal weight of sirup should ordinarily be used for the apparent sucrose determination and this should be weighed and not measured with a sucrose or other pipette. The solutions for the polarizations should be acidulated with acetic acid, after clarification with subacetate of lead, but before filtration. Clarification with neutral acetate is to be preferred.

For the usual factory requirements the sirup may be analyzed by the methods described for the juice, except that the portions for the tests must be weighed and not measured.

#### MASSECUTES AND MOLASSES

**293. Determination of the Density.**—The determination of the density of massecutes and heavy molasses presents certain difficulties which cannot readily be avoided and which compel the acceptance of numbers that are but more or less close approximations, according to the methods used.

As has been explained, the degree Brix of a solution is the percentage by weight of the pure sugar dissolved in it. But in the sugar industry it is usually considered to be the percentage by weight of solid matters, whether sugar or not, in solution. This implies that the solids other than sugar, the non-sucrose, are of the same specific gravity as cane sugar. This is practically true so far as regards the carbohydrate bodies, but is not for the inorganic salts which are associated with the sugars in the massecutes and molasses. These salts having such high specific gravity, as compared with the carbohydrates, influence the density determinations in a very marked degree.

Since the ratio of non-sucrose to the sucrose increases with each stage of the manufacture, as commercial sugar is removed, the difference between the apparent percentage of total solids, as indicated by the hydrometer, and the true percentage, as ascertained by actually drying the material, becomes larger. It is evident from these remarks that calculations of the degree Brix or

apparent total solids in massecuites and molasses, from the density of the product, must be accepted with caution and then only for comparative purposes when similar conditions of analysis are maintained.

There is another condition that has not yet been mentioned. The density (Brix) of a solution calculated from spindling at one dilution is different from that calculated from the hydrometer number ascertained at another dilution. Thus, for example, if one part of a final molasses be dissolved in two parts of water and this solution be spindled, the Brix of the molasses calculated from this spindling will be higher than it would be had one part of the molasses been dissolved in only one part of water. This difference is partly due to the contraction of the solution of sugar on dilution with water and partly to a similar contraction of the solution of the salts in the molasses. This difference would be observed even though one were dealing with a pure sucrose solution instead of molasses. Obviously massecuites and molasses are too dense to be directly spindled, hence one must accept numbers obtained by dilution and spindling that are at best only comparative. The true solids of a final molasses may be from 5 to 10 per cent below the numbers indicated by dilution and spindling.

The methods of dilution and spindling, given in this book, are those customarily used, and the results must not be considered absolute, but only as suitable for comparisons.

**294. Apparent Degree Brix. "Double Dilution" and Spindling.**—Dissolve a weighed quantity of the material in an equal weight of distilled water. Transfer a portion of the solution to a cylinder and determine its degree Brix. Correct the degree Brix for the temperature error as described on page 254, and multiply the corrected number by 2 to ascertain the degree Brix of the material. This is the customary commercial and factory method. The true Brix or percentage of solids is usually several degrees lower than the apparent number obtained by spindling this 1 : 1 solution of the massecuite or molasses.

**295. True Brix or Total Solids by Drying.**—This may be determined by drying on pumice stone or on quartz sand (247) preferably under vacuum and at a temperature of 70° C. Many sugar laboratories use these methods at boiling-water temperature and atmospheric pressure. The heating period is usually fixed arbitrarily (say five hours) as constant weight at this pressure and temperature is not attainable with low-purity materials because of the presence of levulose.

The Spencer electric oven may be used conveniently for these materials (248). There are indications that the very rapid evaporation in this oven may keep the material at a relatively low temperature until drying is nearly complete, when levulose and other organic materials are not so readily decomposable. Moisture in strained honey (which is very high in levulose) was determined in this oven<sup>1</sup> with no appearance of decomposition after twenty minutes heating.

**296. Apparent Brix or Solids by Refractometer.**—The method of using the refractometer has been described on page 264. When dilution and clarification of the sample are unnecessary this method gives results that closely approximate those by drying.

<sup>1</sup> Meade, Ind. Eng. Chem., Vol. 13 (1921), No. 10.

If the material contains crystals of sugar these must be dissolved, since the refractometer only indicates the solid matter that is in solution. When solution is employed the calculation is made as is indicated farther on in this paragraph. Dilution methods involve the contraction error of similar methods by spindling. The error may be reduced by working with very concentrated solutions. If a volume of water be added to a molasses solution, for example, the volume of the mixture is not the sum of the volumes, but is a smaller number, and the concentration is higher. It is, therefore, necessary as in hydrometer methods that the same definite conditions be observed that the results may be comparable.

In the event of testing a highly colored material, the method of Tischtschenko may be used. Mix the material with an equal weight of a solution of pure sucrose of known composition and of as high concentration as is practicable and determine the refractive index. Ascertain the percentage of solids in the mixture by means of the table on page 478. The percentage of solids in the material is ascertained by deducting the per cent of solids in the sucrose solution from twice the solids in the mixture. The principle of the method of calculation for other mixtures is the same as that of the dilution formulæ.

If necessary to dilute the material with water, the calculation is made as follows:

Let  $x$  = the required percentage of solids (Brix);

$W$  = weight of the material used;

$w$  = weight of the diluted solution;

then

$$Wx = bw \text{ and } x = bw/W.$$

**297. Specific Gravity.** *Sidersky's Method.*<sup>2</sup>—This method is applicable to molasses, but not to grained strikes.

The apparatus required is a 50-cc. sugar-flask, a suitable heating arrangement, and a funnel with a glass rod for a stopper. Grind one end of the glass rod with moistened emery into the funnel, to form a stopper. Fill the funnel with a sample of the molasses, and set it in a cylindrical iron support, and heat carefully with the flame of a lamp. The object of the iron cylinder is to distribute the heat. When the material is quite warm, lower the stem of the funnel into the flask, lift the glass rod and fill the latter to within 2 or 3 cc. of the mark. Remove the funnel very carefully so as not to smear the neck of the flask with molasses. Keep the sample hot a few minutes to facilitate the escape of the air, then cool it to the room temperature by immersing the flask in cold water. Dry and weigh the flask and contents, then reduce the temperature of the material to  $17\frac{1}{2}^{\circ}\text{C.}$ , and run water into the flask on top of the molasses, to the mark. The calculations are shown in the following example:

Weight of flask and molasses.....	91.570 grams
Weight of flask empty.....	25.275
Weight of the molasses.....	66.295 grams

<sup>2</sup> *Zeitschrift*, 1881, p. 192.

Weight of flask, molasses and water.....	94.672 grams
Weight of flask and molasses.....	91.570

Weight of water (also its volume in cc.)..... 3.102 grams;

and  $50 - 3.102 = 46.898$ , the volume of the molasses;  $66.295 \div 46.898 = 1.4136$ , the required specific gravity at  $17\frac{1}{2}^{\circ}$  C. By means of the table, page 482, we find the degree Brix corresponding to this specific gravity to be  $79.6^{\circ}$ .

It is very difficult to make a correct test by this method on account of the air-bubbles and as will be shown in the method of Newkirk (succeeding paragraph) the heating causes considerable decomposition and consequent reduction of density. Using no heat, Sidersky's method is of value for determining weight per gallon of commercial molasses as the sample is received.

*Newkirk's Method.*<sup>3</sup>—The difficulties met in the accurate determination of the density of molasses are due to (1) high viscosity, (2) included gases and (3) dissolved gases.

"As a result of the high viscosity the use of hydrometers is prevented, and the thorough mixing or intermingling of the various components of the batch or sample of molasses is made difficult. Obviously, in materials of this character a representative sample can not be obtained without vigorous agitation, which causes entrainment or inclusion of large amounts of air. Finally, the viscosity retards the escape of the entrained or included air. The effects of included air upon the density of the liquor are too evident to need discussing. The removal of the dissolved gases before taking the density may in some cases be essential and in others not.

Investigations at this Bureau established the fact that the removal of gases was best effected by the use of a vacuum. In order to permit this to be done as readily and effectively as possible, an improved pycnometer was designed.

It consists of a bottle, *C* (Fig. 116), fitted with an enlargement at the top, *B*, ground optically flat, and closed with another optical flat, *A*. An expansion chamber, *D*, is ground to the bottle and fitted with a vacuum connection, *E*. To avoid loss of water due to evaporation under reduced pressure, the connecting tube is fitted with a stop-cock, *F*, so that when the proper vacuum has been reached the apparatus can be closed off from the vacuum source. With this provision the volume to be filled with water vapor is very small and the amount of water evaporated will be negligible. The bottle is so shaped as to have a smooth gradual slope to the top, so that the bubbles will rise with the least effort to the expansion chamber. The joints of the expansion chamber, vacuum connection, and stop-cock are ground to an accurate fit.

In using the pycnometer, the expansion chamber, after lubrication of all joints with molasses, is placed on the bottle. The molasses to be analyzed is flowed into the bottle and into the expansion chamber until the latter is one-third full. The vacuum line is then connected and the pressure reduced until the gas expands to visible bubbles. The apparatus is immediately closed off by turning the stop-cock, *F*, and the whole is placed in the thermostat for accurate work or in the balance case for control work. When the bubbles have all collected in the expansion chamber and the temperature has reached equilibrium, the volume



FIG. 116.  
Pycnometer  
for Molasses.

<sup>3</sup> Bur. Stan. Tech., Paper No. 161.

is fixed with the plate after removing the expansion chamber. It is then wiped and weighed.

The densities are determined by correcting weights to vacuo and comparing to the weight of an equal volume of water at 4° C. in vacuo. They are reported in degrees Brix, according to Table 31, on page 459.]

The use of heat was found to cause considerable decomposition of an unknown character, the resulting densities being lower than the unheated sample. The individual accidental error is within one-tenth of a degree Brix, and therefore considerably better than hitherto attainable. The general run of determinations will check to within a few hundredths of a degree Brix.

**298. Weight per Gallon of Molasses.**—This is a figure of great commercial value since molasses is sold both by weight and by the gallon and the relation-

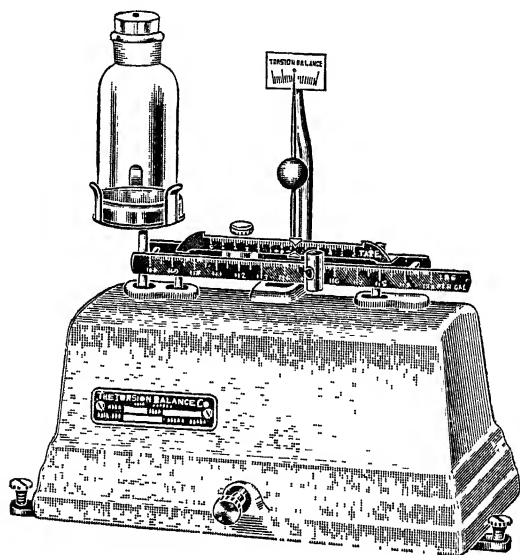


FIG. 117.—Balance for Weight per Gallon of Molasses.

ship between the two is frequently desired. The torsion balance shown in Fig. 117 was designed by H. J. Bastone for determining the weight per gallon. It is equipped with a beam for taring the weighing bottle and a second beam graduated in pounds per gallon from 10.80 to 12.05 in 1/100 pound intervals. In an investigation of this instrument Snyder and Hammond<sup>4</sup> found it more accurate and convenient to fix the volume by sliding a glass disk over the flat polished top of the weighing bottle rather than by wiping off the stopper. After filling the bottle the stopper was inserted and the glass disk was carefully slid over its top surface, thereby removing the surplus molasses. By this procedure only a very thin film of molasses remained between the disk and the top of the stopper. The calibration of the volume of the bottle as well as the direct determinations were made in the same manner.

<sup>4</sup> Bureau of Standards Technologic Paper No. 345.

Their conclusion was as follows:

The torsion balance seems entirely satisfactory for most determinations of weights per gallon of molasses provided the usual precautions are taken to allow the foam to subside and occluded gases to escape. It is obvious that the same precautions must be taken in any method of direct determination of density or weight per gallon of molasses. Any direct reading balance will be found useful in commercial work and routine testing as compared with other methods since results may be obtained without resort to the tedious calculations necessary in a density determination.

Because of entrained air, heavy molasses will never show an actual weight per gallon approaching that calculated from the density obtained in the laboratory. A large series of carefully controlled tests by Wm. B. Saladin at Constancia, Cuba, showed that the weight per gallon of recently pumped Cuban molasses is approximately 86 or 87 per cent of the air-free weight as determined from the density. These figures were obtained on molasses of about 88 apparent Brix, unchanged as it comes from the centrifugal machines. Settled Cuban molasses of the same type will weigh about 11.50 to 11.70 pounds per gallon. Molasses that has been slightly reduced with water and heated before pumping to storage tanks will show weight per gallon figures much closer to those obtained from the true density. Refinery barrel sirups stored for considerable time in large tanks and filled into tank cars by gravity and allowed to settle twenty-four hours before measuring the gallonage average about 11.85 pounds actual weight per gallon as against an air-free weight of 12.15 determined in the laboratory. This same type of material pumped into tank cars shows an average of 11.72 pounds, the difference being the entrained air due to pumping.

**299. Weight of a Unit Volume of Massecuite.**—A modification of Sidersky's method described in 279 may be used to ascertain the weight of a certain volume of massecuite. The selection of the unit volume will, of course, depend upon whether the cubic foot or gallon or metric measures are used.

A device for making this measurement is shown partly in section in Fig. 118. This consists of a cylindrical vessel of any convenient size and preferably of metal. The rim of the cylinders should be ground true, a strip of metal,  $CC'$ , should be provided, which extends from side to side of the cylinder and supports a glass tube drawn to a capillary, as shown in  $TT'$ . Pins  $PP'$  in the rim of the cylinder and fitting in corresponding holes in the strip of metal insure replacing the latter always in the same position. Fill the cylinder to approximately the point  $w$  with massecuite, place the capillary tube in position, then run in water from a burette very cautiously until the tube is reached. The instant the water reaches the tube it rises some distance into it by capillarity. This may readily be noted and more plainly if colored water is used. A previous determination of the volume of the cylinder to

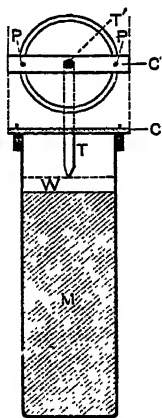


FIG. 118.  
Apparatus for  
Weighing a Unit  
Volume.

the bottom of the tube should be made with water. With this volume and that of the water required to complete the volume with the massecuite, that of the massecuite is readily ascertained and may be compared with the weight of the material.

In making this test the massecuite should be of the temperature at which the measurements of this product are to be made in the factory, and correction for the expansion of the cylinder should be applied.

It is evident that this method cannot be used where vibrations of machinery are felt.

**300. Routine "Purity" Determinations.**—For the ordinary purposes of the factory, for the control of the various processes of manufacture, especially the vacuum-pan and crystallizer work, the absolute percentage of sucrose is not required. The relation, however, between the apparent per cent sucrose and the degree Brix (the coefficient of purity), is frequently needed, but not with a great degree of absolute accuracy. The most important point in connection with this work is to adopt certain conditions of analysis and adhere to them with all similar materials, in order that the results may be comparable.

The ordinary "water purity" which is so generally used in refinery practice, is applicable for this purpose. Dissolve the massecuite or molasses in water and dilute the solution to any convenient Brix between 15 and 20°. Mix thoroughly, allow to stand until all air is removed (or use vacuum connection as described on page 255 to aid air removal) and determine the Brix with a standardized hydrometer, making the temperature correction and hydrometer correction as directed in (243). After Brixing add to a portion of the solution (measured approximately) sufficient Horne's dry lead subacetate to clarify; then add a spoonful of dry kieselguhr and filter through a rapid filter paper. To 50 ml. of filtrate in a 50–55 ml. flask add dilute acetic acid until the contents of the flask are acid to litmus, then make to the 55 ml. mark. Polarize in a 200 mm. tube and add one-tenth to the reading to correct for the extra dilution. If the solution is so diluted that the corrected Brix falls between 12° and 20° Horne's expanded table on pages 493–511, may be used to obtain the coefficient of purity. If the corrected Brix does not fall within the range of the table the purity may be calculated by using the table of factors calculated by Rice from Casamajor's formula modified for hydrometers standardized at 20° C.

In a modification of the above, which obviates acidification and one-tenth dilution in a separate flask, the lead-salt is added as directed above and well shaken to make sure that the clarification is correct. Then dry powdered oxalic acid is added a little at a time until the leaded solution is faintly acid to litmus, after which kieselguhr is added, the whole is thoroughly mixed by violent shaking and filtered. The filtrate is polarized in a 200-mm. tube and the purity determined as before.

The coefficient of purity is all that is usually required in these tests. As the amount of lead used has considerable effect on the purity of low-grade material it is well to use the same amount for tests on similar grades. For higher purity materials than molasses and factory massecuites no acidification is necessary. (See directions for the use of Horne's Table, p. 493.) It should



be noted that because of the large dilution of the material the coefficient is lower than when determined by "double-dilution" methods.

**301. Determination of Sucrose (Polarization).**—For the purpose of comparing the commercial sugar produced in the various periods, or "runs," of the crop as well as determining the total loss of sucrose it is essential that the final or commercial molasses samples be analyzed to obtain correct results as nearly as the processes of analysis will permit. Both apparent sucrose ("polarization") and true sucrose (Clerget) are required for these records. The best and most convenient method is to make these determinations on the 1 : 1 sample made up for the Brix by hydrometer (294). Average daily figures for the apparent Brix, sucrose and purity of the various grades of massecuites and molasses (besides final molasses) are also generally recorded and these are determined by the same methods.

For the apparent sucrose, weigh one normal weight of the 1 : 1 solution into a 200 ml. flask (or one-half normal weight of undiluted material if preferred) add a minimum of 54 Brix lead subacetate solution to effect good clarification, make to the mark, shake thoroughly and filter. Place 50 ml. of the filtrate in a 50–55 ml. flask and acidify with dilute acetic acid making to the mark with water. A solution of acetic acid of such strength that 5 ml. will just acidify the 50 ml. of filtrate may be prepared for routine tests. The amount of lead for a given grade of material need not be varied greatly, if at all, from day to day, and for routine work it is well to prescribe limits for the quantity to be added because of the effect of an excess of lead on the results. Polarize the acidulated solution in a 200-mm. tube and multiply by 4.4 for apparent sucrose.

For dark-colored final molasses the normal weight of the 1 : 1 solution in a 300-ml. flask should be used in which case the polariscope reading is multiplied by 6.6. This multiplication increases any error in reading but it is more accurate than attempting to read a darker solution such as would be obtained with less dilute material.

**302. True Sucrose by Clerget.**—This figure is rarely determined on any low-grade material except final molasses, so the directions here given are for that grade only. Any of the three Clerget modifications, Herzfeld's (224), Steuerwald's (225) or Jackson-Gillis Method IV (236) may be employed, but the latter is preferable. If either the Herzfeld or the Steuerwald methods are chosen weigh two normal weights of the 1 : 1 solution (or one normal weight of the original molasses if preferred) into a 300 ml. flask. Add sufficient lead for clarification, then alumina cream, make to the mark with water and filter. Delead the filtrate with pulverized dry sodium or potassium oxalate. Then filter and proceed with the directions as given under the method selected (224, 225). The direct and invert polarization readings should be multiplied by 6 to give *P* and *I* for the calculations.

For the Jackson-Gillis method weigh out the same amount of molasses as for the other methods (i.e., two normal weights of 1 : 1 solution or one normal weight of the original) in a 300-ml. flask, make to the mark and mix. Pour the solution out in a cylinder, add sufficient Horne's dry lead to clarify, avoiding an excess. A weighed amount can be prescribed for routine work. Mix

by violent shaking and filter. Deleuding is not essential. Pipette two 50 ml. portions of the filtrate in two 100-ml. flasks and proceed as directed in 226.

Polariscopic readings (both direct and invert) should be the average of several concordant observations. Multiply these average readings by 6 to obtain the values for  $P$  and  $P'$ . Divide  $P - P'$  by 3 (the solution weighed out originally was one-third normality) and select the value so found in the table on page 524, opposite which will be found the factor for that concentration. Deduct the temperature correction as found in the same table from the factor and calculate as follows:

$$\frac{100(P - P')}{F} = S.$$

**303. Glucose Determination.**—The amount of molasses or massecuite taken depends upon the percentage of glucose present. Convenient weights for final molasses are 5 grams in 500 ml. if the glucose exceeds 26 per cent; 7.5 grams to 500 ml. for glucose between 18 per cent and 20 per cent, and 10 grams to 500 ml. for glucose between 18 per cent and 10 per cent; giving respectively 0.25, 0.375 and 0.5 gram in the 50 ml. to be used for the test. Prepare the solution by adding kieselguhr and 0.25 gram dry sodium oxalate after making to the mark. (See 230). The addition of the oxalate is recommended to remove the lime-salts which lower the copper reducing power of the glucose. Filter and take 50 ml. of solution, determining the glucose gravimetrically by the Herzfeld method (233). The copper precipitate may be oxidized to cupric oxide, or reduced to metallic copper by the Vorticek-Wedderburn method (page 243), or determined in both ways as a double check. The percentage of glucose is found by reference to Rice's Expanded Meissl-Hiller Tables, p. 512.

The solution may also be prepared by taking double the proportions indicated above, clarifying with neutral lead acetate (3 ml. for each 10 grams of molasses) filtering and deleuding and decalcifying an aliquot portion of the solution with 10 ml. of the mixed solution of disodium phosphate and potassium oxalate recommended by Cook and McAllep (page 239). This method will remove any reducing non-sugars precipitable by lead.

The first method should be used if the method described on p. 316 for lead-preserved juices is employed. Harris<sup>5</sup> proved that deleuding the unfiltered lead-preserved juice with oxalic acid as prescribed gives results which conform with untreated juice. In other words, the total reducing substances are determined in the juice, and for purposes of the "glucose balance" the same procedure would appear to be correct for the molasses and the sugars. If the method of Cook and McAllep is used for molasses it should also be used for the juices, which precludes the preservation of the juice samples with dry basic lead.

The Eynon-Lane volumetric method (239) may be used with either method of preparation of the solution, provided the lime is removed. The concentrations recommended for the gravimetric determinations will serve for the volumetric also.

**304. Determination of the Ash.**—Using 3 or 4 grams of molasses (accurately weighed) proceed with the sulfated ash method as described in 251. The deduction of one-tenth for the sulfating may be omitted if the ash in all products is calculated in the same way. (See page 270.)

**305. Acidity and Alkalinity. Qualitative Tests.**—Solutions of massecuites and molasses are usually so dark-colored that the usual tests for acidity or alkalinity cannot be made. Buisson<sup>6</sup> advises the following method: Transfer 25 cc. of a solution of the material to a glass-stoppered flask; add one drop of neutral corallin solution and 10 cc. of washed ether. Agitate thoroughly and then wait a few seconds for the ether to separate and rise to the surface. The slightest excess of acid or alkali in the ether reacts upon the corallin and changes its color to a yellow or red as the case may be. The water used in dissolving the material must be distilled and the ether must be neutral.

In the experience of Dr. Spencer the success of this method depends largely upon the quality of the corallin. He uses the alcohol soluble corallin as prepared for staining in microscopy. Instead of one drop of the solution he uses several drops of the corallin dissolved in alcohol.

**306. pH Determinations.**—Colorimetric pH determinations are out of the question on dark low-grade products such as molasses or solutions of massecuites. Electrometric methods may be employed but in general the results are not of interest except in special investigations.

**307. Estimation of the Crystallized Sugar in Massecuite.**—*Karcz Method.*<sup>7</sup>—This method, as applied to raw sugar, will be first described, then its application to a massecuite: Weigh 30 to 50 grams of raw sugar and transfer it to a glass dish containing an equal weight of pure anhydrous glycerine. Mix the sugar and glycerine intimately with a glass rod, and place the dish in a desiccator over fused calcium chloride or strong sulphuric acid. Repeat the mixing from time to time until the crystals are well separated and the molasses uniformly distributed in the glycerine solution. This preparatory work requires fifteen minutes and upwards. Place a plug of dry filtering-cotton in the funnel of the apparatus shown in Fig. 119; transfer the mixture to the funnel and replace the cover. Filter off the glycerine solution, using a filter-pump. The mixture should be protected from the moisture of the air during filtration by a calcium chloride tube, as shown at the top of the funnel-cover in the figure.

Since the anhydrous glycerine absorbs moisture with great rapidity, its contact with moist air should so far as possible be avoided.

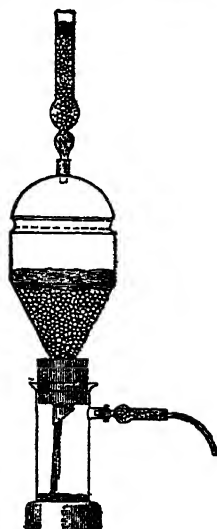


FIG. 119.—Karcz's Apparatus for Crystal Content of Massecuites.

<sup>6</sup> Bulletin de l'Association des Chimistes de France, 9, 597.

<sup>7</sup> Zeit. Rübenzucker-Industrie, 31, 500.

Polarize the normal weight of the glycerine filtrate as obtained above and calculate the crystallized sugar by the following formulae:<sup>8</sup>

$x$  = sucrose in the molasses attached to the crystals;

$P$  = per cent sucrose in the raw sugar;

$p$  = per cent sucrose in the glycerine filtrate;

$$x = \frac{200 - P}{100 - p} p \text{ and } P - x = \text{the percentage of crystallized sugar.}$$

**EXAMPLE.**—Polarization of the raw sugar = 95.6; polarization of the filtrate = 6.75.

$$x = \frac{200 - 95.6}{100 - 6.75} \times 6.75 = 7.55; \text{ and } 95.6 - 7.55 = 88.05$$

the percentage of crystallized sugar.

In view of the large proportion of glucose usually present in cane products, to apply the method to massecuites the sucrose  $x$ ,  $P$ , and  $p$ , in accurate work, should be ascertained by the modified Clerget method, page 231. This is not the case in the following modification of Karcz's method by Pereplechikow:<sup>9</sup>

Transfer the normal weight of the massecuite, treated with an indefinite quantity of anhydrous glycerine, as described above, to Karcz's apparatus and filter off the glycerine solution. Wash the crystals with repeated portions of glycerine until the filtrate is no longer colored. Remove the funnel from the apparatus and wash the crystals into a sugar-flask and polarize them. The polariscope reading is the percentage of crystals in the massecuite.

*Dupont's Method.*<sup>10</sup>—This method usually requires double polarization with cane products.

Heat a quantity of massecuite of known polarization, for example 500 grams to 85° C., and purge the sugar in a small centrifugal, such as is shown in Fig. 130. The sieve of the centrifugal should be covered with thin flannel. Dry the sugar as thoroughly as possible by means of the centrifugal. Polarize the massecuite, the crystals and the molasses. Calculate the percentage of crystals by the following formula:

Let  $x$  = the weight of crystallized sucrose in one part of massecuite;

$a$  = polarization of the massecuite;

$p$  = polarization of the crystals;

$p'$  = polarization of the molasses.

$$\therefore x = \frac{a - p'}{p - p'} \text{ and } 100x = \text{the crystallized sucrose in 100 parts of massecuite.}$$

<sup>8</sup> Zeitschrift j. Zuckerindustrie Bohem, Jan., 1895.

<sup>9</sup> Zapiski, 1894, 18, 346. Bul. Association des Chimistes, 12, 407.

<sup>10</sup> Manuel-Agenda des Fabricants de Sucre, Gallois et Dupont, 1891, p. 293.

EXAMPLE.

Let  $a = 84.5$ ;

$p = 100$ ;

$p' = 60.6$ .

$\therefore x = \frac{84.5 - 60.6}{100 - 60.6} = 0.6066$  and  $100x = 60.66$ , the percentage of crystals in the massecuite.

## CHAPTER XXVIII

### ANALYSIS OF SUGARS

**303. Polarization.**—Weigh the normal weight of the sugar in a nickel capsule. Add sufficient water to moisten the sugar, waiting a moment for the water to penetrate the mass. The moist sugar may usually be poured slowly into a narrow-neck 100-ml. flask without difficulty. A little practice is necessary to accomplish this expeditiously. If difficulty is experienced, a special funnel of nickel (*see* page 213) should be inserted and extend just into the body of the flask. The sugar may be readily washed through the funnel. The capsule, funnel and neck of the flask must be washed with a jet of water. Care should be observed not to use more than about 60 ml. of water in these operations. The flask should be well cleaned before use (*see* page 216) to prevent water from adhering to the neck. Dissolve the sugar by imparting a rotary motion to the flask. Hold the flask above the level of the eye occasionally to see whether all the crystals are in solution. It is essential that no sugar be left undissolved before proceeding to the clarification. A mechanical dissolver should be part of the equipment of all laboratories doing many polarizations. The Multer dissolver shown on p. 213 serves very well.

Having dissolved the sugar, add from 0 to 8 ml. of subacetate of lead (54.3°) the quantity depending upon the grade of the sugar. White sugar requires no lead, but should usually receive a little alumina cream to facilitate filtration. High-grade centrifugals require from 1 to 2 ml. and low sugars, according to their grade, up to about 8 ml. of the lead solution. After mixing the sugar and lead solutions add about 2 ml. of alumina cream and complete the volume to 100 ml., washing down the neck of the flask. If foam interferes with this operation it should be broken down with a drop of ether. The water should be of the temperature of the polariscope room and the flask should be held by the upper part of the neck during the manipulations, to prevent warming the solution. If drops of water adhere to the neck of the flask they should be absorbed by a strip of filter-paper.

Having finished these operations, cover the mouth of the flask with the thumb and mix the solution thoroughly by violent shaking. Pour the entire contents of the flask upon a folded filter in a stemless funnel. Rinse the receiver with the first portions of the filtrate and in all reject fully 25 ml. of the filtrate.

Hardin and Zerban<sup>1</sup> have shown that with perfectly dry filter paper (which is prescribed by the International Commission for Uniform Methods of Sugar Analysis) there is an adsorption of water from the sugar solution which may cause an increase in polarization of more than 0.1 V. By rejecting a full

<sup>1</sup> The Planter. Vol. 73 (1924), No. 20.

25 ml. of the filtrate this adsorption effect is largely, if not entirely, avoided. If very moist paper is used the results may be lowered by the water given up by the paper to the solution. This rejection of the first quarter of the filtrate is mandatory in the Official Methods of A. O. A. C. If the solution is not running clear after 25 ml. have passed through the paper the test should be discarded. The filtration will be rapid and the filtrate bright if the proper amount of lead subacetate has been used. Should the filtrate be cloudy, the whole operation should be repeated, changing the quantity of lead rather than attempt to clear the solution by refiltration. The funnel should be so large that the paper will not project above it and during the filtration funnel and paper should be covered with a clock-glass.

The polarization is made as usual. The polariscope should be tested with a standardized quartz plate before and after use, and the reading should be corrected if need be. The cover-glasses should be carefully selected and be free of flaws and scratches. The sections 219, 221, relative to the influence of clarifying agents and temperature, should be consulted.

Dr. W. D. Horne's dry-lead method may be used with high-grade sugars. After dissolving the sugar, dilute the solution to 100 ml. and add sufficient dry subacetate of lead for clarification, carefully avoiding the use of more than is necessary. Also add a little dry sand to break up particles of lead precipitate that might occlude sugar solution. Close the flask with the thumb and shake it vigorously to mix its contents. Filter and polarize as has been described.

309. "True Sucrose" by Clerget's Method.—Dissolve 52 grams in a 200 ml. flask. Add 4 to 6 ml. of lead subacetate solution (54 Brix) and 1-2 ml. of alumina cream, make to the mark, shake thoroughly and filter. Delead the filtrate by adding dry sodium oxalate, a little at a time avoiding an excess; mix and filter. Pipette two 50 ml. portions into each of two 100 ml. flasks and proceed with one of the Clerget modifications described in Secs. 224, 225, 226. If the Jackson-Gillis Method IV (which is preferable) is selected the dealding of the filtrate with oxalate may be dispensed with, the 50 ml. portions being pipetted from the lead-clarified filtrate. Calculate the Clerget Sucrose according to the method selected.

The relationship between the Clerget sucrose and the direct (commercial) polarization of a raw sugar depends upon the amount and character of the reducing sugars present. If the levulose and dextrose are present in about equal quantities (that is, if the reducing sugars are in the form of invert sugar) the Clerget will be higher than the direct polarization by about one-third of the percentage of reducing sugars.<sup>2</sup> Expressed in symbols the ratio is  $\frac{S - P}{I} =$

.333. If the levulose is in less proportion the ratio will be lowered and the Clerget and commercial polarization will be closer together. If the levulose is still further reduced the Clerget may be actually lower than the direct polarization. Zerban and Hardin<sup>3</sup> showed that there is a seasonal variation of the  $(S - P)/I$  ratio in the raw sugars received at the New York Sugar Trade Laboratory. Composite samples of all sugars received during each

<sup>2</sup> Browne, *The Planter*. Vol. 61 (1918), p. 202.

<sup>3</sup> *The Planter*. Vol. 77 (1926), No. 6.

week were analyzed; commercial polarization, sucrose by invertase and by Jackson-Gillis Method IV, and reducing sugars were determined. In calculating the  $(S - P)/I$  ratio  $S$  was taken as sucrose by the invertase method and  $P$  the direct reading on the *delead* sample for the invertase method. (The direct reading in the Jackson-Gillis Method IV is always low because of the depressing effect of the added sodium chloride.) The results indicated three different periods in the year, viz., (1) January to April when fresh normal sugars were received and the ratio was 0.291, (2) May, June and July when the ratio was low, probably due to the presence of levulose destroying torulae, and (3) August to December when the ratio rose again, due to inversion of the raws in storage by mold fungi and bacteria. A summary of the relationship for the three periods follows:

Period	Commercial Polarization	Direct Reading for Invertase P	Sucrose by Invertase S	Sucrose J. & G.	I	$(S-P)/I$
Jan. to Apr.	96.37	96.24	96.56	96.65	1.11	0.291
May, June and July.	96.25	96.10	96.25	96.32	1.18	0.139
Aug. to Dec.	95.95	95.75	96.05	96.09	1.36	0.230
Year, 1925.	96.16	96.00	96.28	96.34	1.23	0.230

In the 52 weekly analyses, 42 showed sucrose by invertase higher than the commercial polarization; in one case the two figures were the same, while in the nine other cases the Clerget by invertase was lower than the ordinary polarization.

**310. Determination of Glucose.**—For the ordinary run of 96° raw sugars containing less than 1.5 per cent of glucose, use Herzfeld's Gravimetric Method No. II (Sec. 235). Forty grams of sugar are weighed into a 200 ml. flask, dissolved in water and made to the mark. After mixing well add dry kieselguhr and 0.25 gram (approx.) of dry sodium oxalate (the latter to remove lime salts), and filter; 50 ml. of the filtrate being used for the determination. Clarification may be effected with neutral lead subacetate (about 3 ml.) then filtering, after which the mixed solution of disodium phosphate and potassium oxalate (3 grams + 7 grams: 100 ml.) according to Cook and McAllep (Sec. 230) is used in an aliquot portion of the filtrate to decalcify and delead. The volumetric method of Eynon and Lane (Sec. 239) may be selected, using either method of clarification.

If the sugar is of lower test and contains more than 1.5 per cent and less than 3.5 per cent glucose weigh 20 grams to 200 ml., if more than 3.5 per cent glucose, 10 grams to 200 ml. For calculating the results with the gravimetric method (Herzfeld No. I) use Rice's Tables, p. 513. The above concentrations of solution serve for the Eynon-Lane method also.



**311. Estimation of the Moisture.**—The method of drying sugars in an ordinary oven is described in Sec. 247. As a rule a fixed drying period (3–4 hours) is used in sugar laboratories, rather than attempting to dry to constant weight.

The Spencer electric oven has proved both rapid and accurate for sugar moisture determinations. It is particularly valuable in controlling the percentage of moisture in sugars as produced in order to insure good keeping quality of the raws in storage (see "Factor of Safety," p. 139). The method of drying sugars in the Spencer oven is described in Sec. 248.

**312. Determination of the Ash.**—The sulfated ash method as described in p. 269 is generally used in both factory and commercial testing. Weigh 5 grams of sugar in a platinum or fused quartz dish, add 0.5 ml. of concentrated sulfuric acid, ignite gently until fully carbonized, then at low red heat in a muffle add a few drops more of acid, ignite to constant weight and weigh. Express the result, without deduction, as sulfate ash or deduct one-tenth, depending on the practice employed. (See p. 270 concerning deduction.) The ash may be determined electrometrically as given in 253, 254.

**313. Determination of Gums.**—The percentage of crude gums in a raw sugar is known to affect its working qualities in the refinery, though the determination is not generally included in routine procedure. The results of the determination are dependent upon many factors, and strict adherence to the technique and method is essential for comparable results.

Ruff and Withrow<sup>4</sup> specified the following details after a careful study of the various methods in the literature. Dissolve the sugar in an equal weight of water. If the sample is a solution (e.g., juice or sirup) concentrate or dilute to about 50 per cent of solids. With materials of a high gum content use 5 grams of solids (10 ml. sample); with those of low gum content use 10 grams (20 ml. sample). Sodium benzoate (0.5 per cent) may be used to keep sirup for gum analysis. (Suspended matter in ordinary raws may be disregarded. For juices containing much suspended matter filter through asbestos.)

Add to the sugar solution 0.5 ml. of concentrated hydrochloric acid for the 10 ml. sample, and 1.0 ml. of acid for the 20 ml. sample; then add 50 ml. of 93 to 96 per cent (by volume) alcohol to the 10 ml. sample, and 100 ml. of alcohol to the 20 ml. sample. This precipitation is conducted preferably in beakers of 250 ml. capacity, the alcohol being added from a rather slow delivery pipette, while the mixture is thoroughly and vigorously agitated. The precipitate is allowed to settle for fifteen minutes before filtering, to avoid slow filtering.

The filtration is done through an asbestos mat Gooch crucible, this type of crucible having been found to be superior to alundum for this work. The mat should contain at least 0.20 gram of dry asbestos. It is unnecessary to wash the mat free from fine fibers or to weigh the crucible before filtering the gums. The vessel containing the precipitate should be drained before rinsing, and the rinsings of wash alcohol should not be added before the last drop of original liquor has passed through the mat. The precipitate is then washed with alcohol of the same strength as that used for the precipitation.

<sup>4</sup> Ind. Eng. Chem. Vol. 14 (1922), No. 12.

After washing, the crucible is dried at 100°–105° C. to constant weight, one hour usually being sufficient. The crucible is cooled in a desiccator and accurately weighed on an analytical balance. After drying and weighing, the crucible is ignited to complete combustion of the carbon. The crucible is again cooled and weighed. The difference between the weight after drying and the weight after ignition represents the dried gums. For very accurate work, or where the precipitate to be weighed is very small, a correction must be applied for the loss in weight of the dried crucible and mat during ignition. This correction may be determined once for all for any crucible and mat. The mats are nearly constant in weight if prepared by using the same volume of a standard stock solution of asbestos thoroughly shaken in water for each determination.

The alcohol used for precipitating purposes must be from 93 to 96 per cent by volume. Either pure ethyl alcohol, or alcohol denatured with methyl alcohol alone, is preferable for gum precipitation. All alcohol should be filtered before use. It is advisable to use the same strength alcohol for washing as was used for precipitating the gums. Occasionally a little acid in the wash alcohol will speed up the filtration.

**314. Dye Number.**—This is a method of estimating the relative amount of colloids in a sugar and is described in detail in 152. It is used largely as an indication of the filtrability of raw sugars in the refinery.

**315. Iron in Sugars. Sulphide Colorimetric Method.**<sup>5</sup>—This test is rarely used except in special work. Prepare a stock solution containing 10 grams of pure crystallized ferrous sulphate,  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  dissolved in a 50–60 per cent sucrose solution, with the addition of a few drops of sulphuric acid, and dilute to 1000 ml. with the sugar solution. The acid should be very largely diluted before adding it to the sugar solution.

Dilute this stock solution with distilled water, from time to time as required, *e.g.*, 10 ml. to 100 ml. and 50 ml. of this solution to 500 ml.

The tests are made in Nessler's cylinders, a number of which of the same diameter and height should be provided. Into a series of these cylinders measure increasing amounts of the diluted stock solution, noting the quantity of iron in each, and dilute each to 100 ml. Add a milliliter of recently prepared monosulphide of ammonia to each and stir. Dissolve 3 to 10 grams of the sugar in a Nessler's cylinder, dilute the solution to 100 ml. and add 2 ml. of the sulphide solution. Let the cylinders stand ten minutes and then match the color of that including the sample with one of those containing the stock solution. Both then contain the same quantity of iron, *i.e.*, the quantity of iron in the sugar used. The cylinders should stand on white paper in making the comparisons.

The sulphide is prepared by saturating ammonium hydroxide with sulphuretted hydrogen and then adding an equal volume of ammonium hydroxide. In the case of dark sugars, incinerate the sugar, with the addition of iron-free sulphuric acid, burning at the lowest possible temperature. Dissolve the ash in a minimum quantity of iron-free hydrochloric acid and proceed with this solution as has been described.

<sup>5</sup> Eastick, Ogilvie and Linfield, *Int. Sugar Journ.*, 14, 428.

**316. Color of Sugars.**—The color of raw sugars has become an important figure both for the producer and the refiner. It may be determined in any of the several ways described in the chapter on color determinations, p. 284. The two simplest instruments for routine laboratory use are the Hess-Ives Tint Photometer (263) or some form of Duboscq colorimeter (262). For either instrument prepare the solution as follows:

Weigh 125 grams of sugar into a tared Erlenmeyer flask including stirring rod, then add boiling water a few milliliters at a time, stirring vigorously and constantly until solution is complete, being careful that the total weight of sugar and water does not exceed 200 grams. The brix of the solution must at all times be 60° or above to avoid coagulation of colloids (see p. 285). The solution is allowed to cool and the necessary amount of water is added to complete the weight to exactly 200 grams. Add a weighed quantity of washed and dried kieselguhr and filter repeatedly until the solution is free from turbidity as evidenced by a slanting beam of light against a dark background.

For the Hess-Ives instrument, place this solution in a cell of suitable thickness (3 mm. or 6 mm. depending on color of solution), read through "560" screen, recording as Meade-Harris units (p. 292) and calculate to 1 cm. thickness. Color units are proportional to cell thickness ( $-\log t$  values may be obtained by the use of the Rice scale, p. 293). For any of the Duboscq instruments, place the clear solution in one of the tubes and a standard caramel-in-glycerine solution in the other, set the standard at any convenient figure (say 20 mm.) and compare the length of the test column solution required to match the standard. Calculate the color as described on p. 288.

If dilution of color is necessary this is accomplished by the addition of the requisite amount of a 60° Brix colorless sugar solution (see 285).

It is recognized that this procedure is not of value except for comparative purposes. Kieselguhr has a selective adsorptive action on color but the other methods of preparing optically void solutions are so tedious and open to question (see p. 285) that the kieselguhr is advocated for routine comparative tests because of its simplicity. The methods given above should give results as accurate as are required by the raw sugar producer for his purposes. For special work in conjunction with refinery char-filter studies a complete color analysis by the spectrophotometer is of great value. (See p. 293.)

**317. The Dutch Color Standards.**—Foreign sugars, on entering certain countries, pay duty according to their polarization and color. If their color is No. 16 Dutch standard, *e.g.*, or lighter, they pay a higher rate of duty than if darker than this standard.

The Dutch color standards consist of a set of samples of sugar numbered up to 20, which is white sugar. These are prepared by an establishment in Holland and are supplied the sugar trade in sealed bottles. The samples should be renewed from time to time, since the color of the sugar is not permanent.

Centrifugal 96° sugar will usually be darker than No. 16, especially when molasses is boiled in with the first sugar. The chemist of a tropical sugar factory should be supplied with a sample of No. 16, so as to avoid the shipment of sugar of this color or lighter to certain countries and consequent loss to the factory-owners.

**318. Rendment.**—The rendment is the estimated yield of refined sugar that a raw sugar will produce based on refining experience. With the almost complete disappearance of low-grade sugars from the raw sugar market, the polarization and certain “refining qualities” (see p. 141) are sufficient to determine the percentage of yield to be expected. Formerly, American refineries deducted five times the percentage of ash from the polarization of the raw sugar to obtain the percentage rendment or the “analysis” of the sugar. This practice has now become obsolete.

## CHAPTER XXIX

### ANALYSIS OF THE FILTER-PRESS CAKE

**319. Preparation of the Sample.**—The sample of press-cake obtained as directed in 274, should be reduced to small fragments and mixed by means of a spatula or large scissors. If the cake is very soft it may be necessary to rub it to a paste in a large mortar, then subsample it. With good care and careful management the press-cake will be firm and the sample may be readily reduced.

**320. Moisture Determination.**—Dry 5 grams of the press-cake, in a shallow tared dish, to constant weight in a water-oven at approximately 100° C. The loss of weight  $\times 20$  = percentage of moisture.

The sample should be partly dried at a low temperature before heating the oven to 100° C., otherwise the surfaces of the fragments of press-cake may be covered with a glazed coating which would prevent the escape of moisture.

Moisture may be determined on a large sample in the Spencer electric bagasse oven (323) or in a smaller amount (5 grams) in the oven used for sugar moistures (248). The bottom of the capsule should be covered with asbestos to prevent dust being pulled through the wire-mesh by the air current. Moisture in press-cake is not usually included in the daily routine tests as the appearance of the cake is sufficient guide to the factory superintendents and the pressmen.

**321. Sucrose Determination.**—Transfer 25 grams of filter-press cake to a small mortar. Add boiling hot water to the sample and rub it to a smooth cream with the pestle. Wash the material into a 100-ml. flask with hot water, cool, add 6 ml. subacetate of lead solution (54.3° Brix), complete the volume to 100 ml., mix the contents of the flask thoroughly, filter, and polarize. The polariscope reading is the percentage of sucrose in the press-cake.

It is usually more convenient to use 50 grams of the sample, and add the subacetate of lead while rubbing the material to a cream and wash all into a 200-ml. flask. This facilitates the removal of the last portions of the press-cake from the mortar. A flask with the neck enlarged above the graduation is more convenient in this analysis than an ordinary sugar-flask (Fig. 120).

The object in using 25 grams of the material in this analysis instead of the normal weight is to correct for the volume of the lead precipitate and that of the insoluble matter.

Parallel experiments, on a sample of filter-press cake, by the method

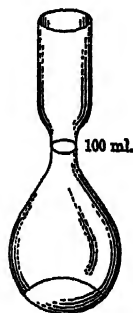


FIG. 120.—Kohlrausch Flask.

described, and also modified by adding acetic acid, after cooling, to decompose saccharates of lime and precipitated levulose, gave in each 9.6 per cent of sucrose. A third portion of this sample was rubbed to a cream with hot water and defecated with subacetate of lead, then washed onto a filter. The washing was continued with very hot water to nearly 200 ml. of filtrate. The filtrate was cooled and diluted to 200 ml. and polarized, giving a reading of 4.6. The residue was washed into a sugar-flask and diluted to 100 cc. and filtered. The filtrate polarized 0.5. The sum of  $4.6 \times 2 + 0.5$  is 9.7, or nearly the same as in the other two experiments, showing that 25 grams is approximately the correct amount of average material to use instead of the normal weight. Many experiments by Dr. Spencer have given similar results to those described.

A routine method devised by Spencer uses an ordinary "milk-shake" shaker to insure disintegration of the sample. Fifty-two grams of the finely divided press-cake are placed in the tumbler of the shaking machine, then 162 ml. of distilled water (room temperature) using an automatic burette. The proportions of press-cake and water are now as above. About 2 grams of Horne's dry lead are added, the tumbler is covered and the machine put in motion to agitate the mass violently. Filter, add two or three drops of concentrated acetic acid to acidify and polarize in a 200-mm. tube.

## CHAPTER XXX

### ANALYSIS OF THE BAGASSE

**322. Preparation of the Samples.**—After securing the sample as described in 267, it should be rapidly and thoroughly mixed and subsampled. Modern milling in long trains and under high pressures disintegrates the bagasse to such an extent that many laboratories make both the sucrose and moisture tests on the sample in the condition that it comes from the mill. Others continue the use of choppers or shredders.

The condition of the sample for the sucrose test is especially important. The pieces should be very small. The Java laboratories require this sample to be fine enough to pass through a 4-mm. sieve. De Haan <sup>1</sup> collaborated in the preparation of the Java instructions for bagasse analysis in which the above specification as to the sample is given. He, however, has stated <sup>2</sup> that the apparent influence of fineness is really largely due to incorrect sampling. This implies that the chemist unconsciously selects the larger pieces rather than an average of the sample. Norris <sup>3</sup> specifies 6 mm. as the diameter of the largest admissible piece of bagasse. Attempts to reduce the material finer than Norris' specification may lead to errors through the drying of the sample, especially when the sucrose content is high. Norris <sup>4</sup> made numerous tests to ascertain the average loss of moisture during the preparation of the sample and found it to average 2 per cent when the chopper is covered and 4 per cent when uncovered. From these observations it is evident that the sample should be prepared as rapidly as is possible, and even then the sucrose numbers may be a little too high. There is often a compensating error that may offset this error, viz., slightly imperfect extraction of the sucrose in the digestion.

Slow preparation of the sample may result in a large error in the moisture test. This may be avoided by drying the bagasse in the condition that it comes from the mills, using the special oven described farther on.

The Warmoth disintegrator, already described on page 307 for use in the analysis of cane, is the best and most rapid method of preparing the bagasse sample for analysis. Experience has shown that about 1 per cent of moisture is lost during sampling, storing and disintegration in this machine. By using 99 grams of the bagasse sample thus prepared both for the moisture and sucrose

<sup>1</sup> Int. Sugar Journ., 1912, 14, 43

<sup>2</sup> Ibid., 5.

<sup>3</sup> Bul. 32, Haw. Sugar Planters' Expt. Sta., 32.

<sup>4</sup> Ibid., 8.

determinations, instead of 100 grams, this evaporation is compensated for, and furthermore the relationship between moisture, sucrose and fiber are strictly comparable since the tests are made on the same sample. This latter point is equally true if both the sucrose and moisture are made on the sample in the condition that it leaves the mills. It is not so certain if the moisture is made on the unprepared sample and the sucrose on a chopped or disintegrated portion.

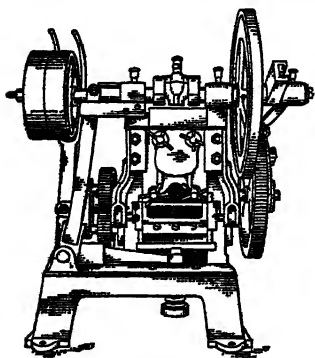


FIG. 121.—Bagasse Chopper  
(Boot and Krantz.)

The Boot & Krantz chopper, Fig. 121, is suitable for preparing the sample. The knife is adjustable to cut the bagasse to any desired fineness. The machine may be driven by belt or by hand power. The Athol meat-chopper, size No. 405, Fig. 122, is an efficient machine for reducing bagasse. This machine has large capacity and may be covered during chopping. Neither of these machines is as rapid nor as thorough as the Warmoth disintegrator.

**323. Determination of the Moisture.**—As has been indicated in the preceding paragraph, it is of importance to dry a large sample on account of the lack of uniform distribution of the moisture.

Experiments upon both a manufacturing scale and in the laboratory have shown that bagasse may be heated to high temperatures without appreciable decomposition. Such tests were made in drying shredded cane at Preston, Cuba, where the temperature employed was very much higher than is here suggested for laboratory work. Drying in vacuum-ovens at low temperatures is not dependable. Many writers recommend drying samples of 20 grams or even less. From the point of view of the test of the particular 20 grams there is no objection to this quantity, but so small a sample cannot accurately represent a material such as bagasse. The methods of drying given in this book are arranged in order of preference.

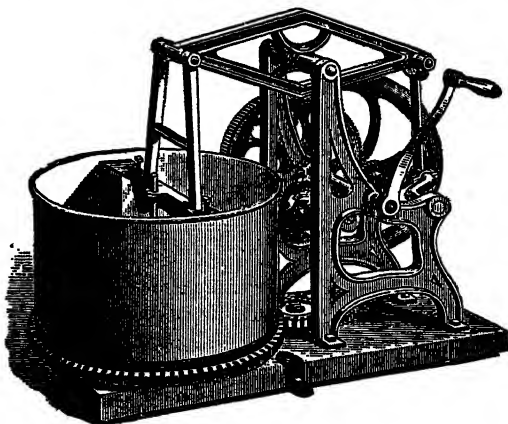


FIG. 122.—Bagasse Chopper (Athol).



*Drying in a Current of Heated Air. Spencer Electric Oven, Large Size.*—This oven, especially designed for the determination of moisture in bagasse, is designated as Type "B" to distinguish it from the smaller oven used for sugars, juices, etc. (Sec. 248). The bagasse oven is essentially the same in principle as the smaller size, being a means of drawing a large volume of heated air through the sample to be dried, the air first passing over an electric heating element. The suction is best applied by connecting with the factory vacuum system. The capsules are 10 cm. diameter by 20 cm. high fitted with a bottom of metal filter-cloth to allow free passage of the air. The test is made on 100 grams of the bagasse as it comes from the mills (i.e., without chopping or otherwise comminuting), or on a disintegrated sample using 99 grams, or whatever amount has been found proper by experience to compensate for the moisture lost in preparation of the sample. This sample, in a tared crucible, is placed in the oven, the suction started and current to the heating element regulated to give a temperature of 135° C. which is continued for forty-five minutes, after which the capsule is cooled and weighed. A simple method of avoiding all calculations is to place the tare and a 100-gram weight on one pan of the balance and the capsule containing the dried sample on the other. Weights are then added to the pans containing the sample, this weight in grams representing the percentage of moisture in the bagasse.

*Special Hot Air Oven.*—This oven, also designed by Spencer, was the forerunner of the electric oven just described. As shown in Fig. 123 it is a cylindrical cast-iron vessel, C, provided with a removable cover, D. A rubber gasket, I, and the clamps, E, insure an air-tight joint between the body of the oven and the cover. The clamp may not be necessary if the door is heavy and the joints are well ground. A steam-ejector, F, draws a very strong current of air through a heater, the pipe G, and the basket A. The air is heated by passing it over a steam-coil. It may receive a preliminary drying by drawing it over quick-lime, followed by filtration, but this does not appear to be necessary. A small size feed-water heater is an inexpensive and efficient heating device. If this heater is used with exhaust steam from the engines, the air should be drawn through the copper coil, the steam surrounding it, but with live steam the air should pass over the coil. The specific heat of air is low, hence the pipes should be covered and a very large volume of air must be used.

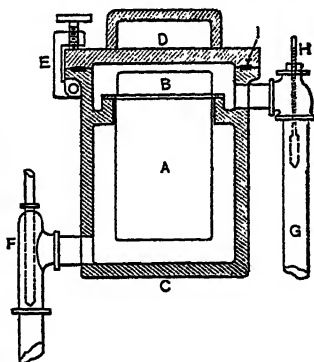


FIG. 123.—Bagasse Oven with Induced Draft.

A cylindrical bagasse-basket of very thin sheet brass, A, fits in the oven as is indicated. Two or more baskets should be provided and tared if many samples are to be dried. The bottom of the basket is of finely perforated brass plate, such as is used in centrifugals. Sheet brass containing about 625

round holes per square inch is suitable for this purpose. The top of the basket is open. A narrow flange supports the basket and makes a joint with the iron castings. The weight of the basket and bagasse and the pressure of the air are usually sufficient to insure a good joint, but if need be, a gasket of steam-packing may be used. The thermometer, *H*, indicates the temperature of the air current. If the oven forms one of a battery, a stop-valve should be provided in the pipe *G*. A valve is also convenient in regulating the current of air. If it is desired to leave the steam turned on the ejector, *F*, when opening the oven, it is necessary to provide an air-cock in the cover or elsewhere for use in breaking the vacuum.

This oven may be constructed of any convenient size. Those constructed with a basket 8 inches in diameter and 12 inches deep will hold 1 kilogram of loose bagasse or 2 kilograms if lightly packed. The drying period is extended a few minutes by the packing. A basket of this size may be used with the 5-kilogram balance shown in Fig. 81.

The method of drying is as follows: Assuming efficient mill-work, reduce the sample to about 2 kilograms by subsampling; fill the tared basket with 2 kilograms of bagasse, packing it lightly as may be necessary. Insert the basket in the oven, replace the cover and turn sufficient high-pressure steam into the ejector to produce a very strong current of air. The air should be heated to any convenient temperature above 110° C. and preferably to 130° C. The air-pipe should be well covered.

The drying period varies with the temperature of the air and the condition of the bagasse. At 110° C. the period is usually about ninety minutes. At the close of the drying-period, which after a little experience with the oven may be arbitrarily fixed, remove the basket by the bail, *B*, and place it in a desiccator to cool. A large earthenware jar will serve as a desiccator. After cooling, ascertain the weight in grams of the dry bagasse and divide this number by 20, to arrive at the per cent of dry matter. The per cent moisture is 100 minus the per cent of dry matter.

The oven as originally designed<sup>5</sup> is self-contained. The lower section contains the heating-coils, and in the upper are six bagasse drying-tubes of 200 grams capacity each. The oven illustrated is less expensive and is more accessible for repairs than the older model. It may easily be built in the plantation shops, largely from old material.

*Drying in Metal Trays.*—Shallow metal trays may be used as containers for the bagasse in conjunction with the oven described in the next paragraph. A suitable size for use on the sugar balance and holding 50 grams of bagasse is 4 inches by 8 inches by 1.25 inches deep. The drying progresses faster if the bottom of the tray is of wire gauze or perforated sheet brass. The temperature of the oven should be at least 110° C. and preferably 130° C. Larger trays may be used if a balance of sufficient size is at hand.

*Steam drying-oven.*—The steam drying-oven is shown in Fig. 124. It is most conveniently constructed of 2-inch planks of well-seasoned lumber. Two steam mainfolds, or coils, *C*, of iron pipe are used with live steam to heat the

<sup>5</sup> Spencer. *Journ. Ind. and Eng. Chem.*, June, 1910, 2, No. 6.

oven. Half-inch iron pipe is suitable for making the coils. The use of two coils, instead of one, facilitates regulating the temperature.

Holes in the door of the oven at *A* admit air, and the moist warm air escapes at *D*. A thermometer can readily be inserted in the oven by boring a hole in the wall near the shelf *B*. This shelf or tray *B* is made of a wire screen. The mesh should be large. The door and other parts should be protected from warping by the usual carpenter's expedients. There should be globe-valves on each of the inlet and tail-pipes, to each coil, to regulate the steam and the discharge of the condensation water.

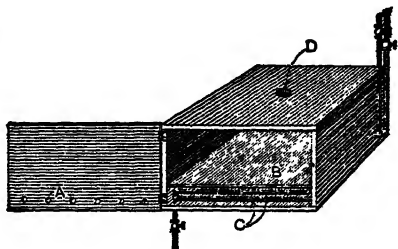


FIG. 124.—Bagasse Oven.

**324. Determination of the Sucrose.—Single Digestion Method.**—This consists, in general, of digesting a weighed quantity of finely divided bagasse (100 grams usually) in a measured quantity of water (say 900 or 1000 ml.) at or near the boiling temperature for one hour in a tared container. Suitable means for agitating the solution and bagasse during the digestion period are usually provided. Five ml. of 5 per cent sodium carbonate solution are

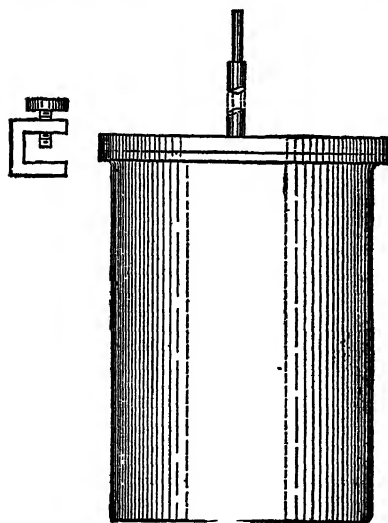


FIG. 125.—Bagasse Digester.

included if the bagasse sample is fresh or was preserved with formaldehyde. If the ammonia-chloroform mixture recommended in Sec. 267 is used as a preservative the soda is omitted. After the digestion period the container is cooled and weighed, the solution drained off, clarified with dry lead and polarized. The calculation of the sucrose percentage is explained in an example given later.

A convenient digester, Fig. 125, is of copper about 4 inches in diameter and 6 inches deep, provided with a brass cover and clamp for making a tight joint. A brass tube attached to the cover serves as a condenser. A brass rod, carrying a small disk for mixing purposes, should pass through and extend above the tube. The rod should

be moved up and down occasionally either by hand or mechanically. A Kodak developing-tank, fitted with a condensing-tube, may conveniently be used as a digester.

No water should be added to the bagasse after starting the digestion. The

boiling should be very gentle or, preferably, the liquid should just reach the boiling-point.

The test is conducted as follows: Tare the dried digester, add 100 grams of bagasse (or 99 grams, or whatever amount has been found to compensate for the loss of moisture during disintegration, as previously explained), then 1000 grams of boiling water (0.25 gram of dry sodium carbonate should be added to the water if ammonia has not been used as a preservative). Close the digester and place in a steam or water bath and heat at the temperature of boiling water for one hour, during which the contents of the container are agitated by means of the rod as described. No water should be added to the bagasse after starting the digestion.

Cool and weigh, drain off some of the liquid (about 150 ml.) place in a cylinder and add sufficient dry lead subacetate to clarify. A little dry kieselguhr may be used to insure a clear filtrate. Shake vigorously, filter and polarize in a 400-mm. tube.

The following calculation illustrates the method of arriving at the percentage of sucrose but this may be dispensed with in practice by using the table on page 525.

Weight of bagasse used.....	100 grams
Fiber in bagasse.....	48 per cent
Weight of digester, bagasse, water..	1375.5 grams
Weight of digester.....	175.5
<hr/>	
Weight of bagasse, water.....	1100 grams
Weight of fiber.....	48
<hr/>	
Weight of extract.....	1052 grams
Polariscope reading in 400-mm. tube	3.2
From Schmitz's Table 3.2 = .....	0.83
0.83 ÷ 2 = .....	0.415 per cent sucrose in thin solution
1052 × 0.415 = 4.37 grams = .....	4.37 per cent sucrose in bagasse.

By the table on page 525: Opposite 3.2 and under 1050 (*W*) is found 4.37. By interpolation for 1052 the per cent sucrose is 4.38 per cent corresponding with the above. The difference of 0.01 per cent sucrose is due to the fact that the figures for tenths in Schmitz's table are averages for a range of degrees Brix and polarizations.

*Spencer Rotary Digester.*—This apparatus was invented by Dr. Spencer with a view of obviating some of the difficulties involved in the use of the ordinary digester. It consists (Fig. 126) of a cylindrical steam bath in which three cylinders are rotated at 5 r.p.m. by means of a small motor and reducing gear. Hand operation is also provided for. The cylinders for the bagasse samples are similar to the digester previously described, being  $4\frac{1}{4}$  inches by 8, with tight covers held in place by screw clamps. Vacuum-breaks are provided in the covers. These cylinders revolve endwise, the slow motion of the shaft

causing the bagasse to fall from end to end in the solution promoting maceration and diffusion. Steam and cold water connections to the casing provide efficient means of heating and subsequent cooling. The test is conducted and the calculation made in the same way as has been previously described.

*Repeated Digestion Method.*—In a suitable dish, preferably a porcelain casserole, cover 100 grams of finely divided bagasse with water and boil it during ten minutes. Drain off the liquid, pressing the bagasse with an iron spoon. Repeat this digestion with water and the decantation in all eight times. Press the residue of the bagasse in a powerful hydraulic or other press and unite the liquid expressed to those portions already drained off. Cool the liquid to the ordinary temperature and measure it, adding, if need be, a little water to bring it to an easily measurable volume. Clarify the solution by Horne's dry-lead method, page 314. The degree Brix of this very dilute solution may be neglected. Polarize the solution, using a 400-mm. observation-tube, and calculate the sucrose with the aid of Schmitz's table on page 481 dividing by 2 to compensate for the tube length. This per cent sucrose is that of the extract. Consider the cubic centimeters of the extract to be grams and multiply this number by the per cent sucrose to ascertain the per cent sucrose in the bagasse. About 0.5 gram of carbonate of sodium should be added to the water used in making the first extraction.

Geerligs<sup>6</sup> states that long digestion with boiling water dissolves a dextro-rotatory gum from the fiber of the bagasse and makes the polarization too high. De Haan extracted this gum and found that it is precipitated by the subacetate of lead.<sup>7</sup> Norris failed to find this dextro-rotatory substance in extracts from Hawaiian cane.<sup>8</sup>

*Norris' Method.*<sup>9</sup>—Dr. R. S. Norris, noting that one of the weak points in the sucrose test is the small polariscope reading, devised the following modification of the single-digestion method: A special digester, Fig. 127, A, is used.

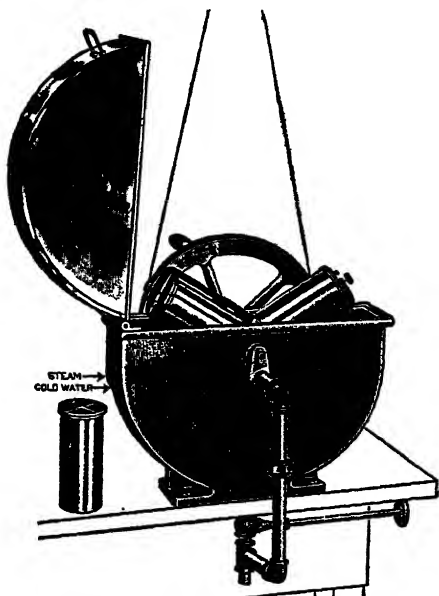


FIG. 126.—Spencer Bagasse Digester.

<sup>6</sup> Int. Sug. Journ., 11, 156.

<sup>7</sup> Bul. 32, Haw. Sugar Planters' Expt. Sta., 12.

<sup>8</sup> Ibid., 18.

<sup>9</sup> Bul. 32, Haw. Planters' Expt. Sta., 24; Int. Sugar Journ., 12, 641.

This is like the double cooker used in the kitchen for cereals, etc. A tamp, *C*, made of a heavy perforated metal disk and provided with a substantial handle, is fitted rather snugly into the inner vessel, *B*, and serves to press the bagasse and also as a cover. The inner vessel should be 4.25 inches deep and 4.25 inches in diameter.

Weigh 100 grams of bagasse in the tared inner vessel. The tamp should be tared with the vessel. There should be no pieces of bagasse larger than 6 mm.

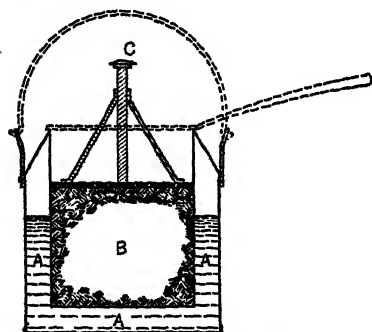


FIG. 127.—Norris Bagasse Digester.

in diameter. Add 500 ml. of hot water containing 5 ml. of a 5 per cent solution of carbonate of sodium. Place the vessel in the boiler and digest for one hour, pressing the bagasse and mixing the solution every fifteen minutes. The tamp should be fastened at the top of the inner vessel, when not in use, and serve as a cover. It should not be removed until after the completion of the digestion and weighing. At the conclusion of the digestion period, remove and wipe the inner vessel and set it aside to cool and weigh, or it may be

cooled by placing it in cold water. The calculation of the per cent sucrose is made as in the single-digestion method, page 346.

Norris states that the shape and dimensions of the vessel apparently influence the results. The vessel should not be too deep. The dimensions given are those decided upon by Norris after many experiments.

**325. Determination of the Fiber (Marc).—**The fiber may be determined directly as follows if the Spencer oven has been used for the moisture determination: Otherwise the method described for the analysis of cane (280) must be employed. Cover the capsule with a small mesh screen and wash the dried bagasse sample with hot pure water (condensation water) until wash water is free of sugar by alpha naphthol test. Then wash the fiber with two or three liters of boiling distilled water; dry in the Spencer bagasse oven as for moisture determination and weigh. Weight of fiber in grams = per cent fiber in bagasse.

The indirect method is much preferable and direct fiber determinations are now rarely made. This method was adopted by Dr. Spencer after several thousand comparative tests in The Cuban-American Sugar Co.'s laboratories. The required data are obtained in the mill control.

Let *S* = the dry matter in the bagasse;

*P* = the per cent sucrose in the bagasse;

*C* = the coefficient of purity of the residual juice (*see* next section);

*x* = per cent fiber (marc) in the bagasse,

then

$$x = S - 100P/C.$$

Steuerwald, of the Java Experiment Station, in an investigation of the various methods of fiber determination, arrived at the conclusion that the indirect method as above gives the most reliable results. He considers the claims of the water- and alcohol-extraction methods and concludes that the aqueous methods give high figures and the alcoholic extraction, even correcting for the separation of saccharetin from the fiber, gives low figures.

**326. Purity of the Residual Juice.**—The residual juice, *i.e.*, the juice remaining in the final bagasse, is considered by many chemists to correspond in purity to that of the juice extracted by the last mill of the train and experience indicates that this is near the truth. However, since the bagasse receives its final and heaviest pressure between the last pair of rolls, the juice flowing from the last roll probably more nearly approximates the true residual juice. The coefficient of purity of this juice is used in calculating the fiber.

The analysis is made as for other juices.

## CHAPTER XXXI

### ANALYSIS OF FACTORY WASTES

**327. Analysis of Foam from Sirups, etc.**—A current of compressed air, or a little ether rapidly evaporated, will quickly reduce the foam to a liquid. Remove the ether by evaporation over warm water, at a safe distance from fire, and proceed with the analysis by the methods described, beginning 292.

**328. Sugar in the Boiler Feed-water.**—The feed-water for the steam-boilers in a cane sugar factory is largely derived from that evaporated from the juice and sirup. The water from the steam condensed in the calandria of the first vessel of the multiple effect, the calandrias and coils of the vacuum-pans and the coils and tubes of the various heaters forms a very important source of water supply for the boilers. Sugar may enter these waters through entrainment with the vapors from the juice and sirup and through defects that develop in the heating-surfaces.

Sugar causes the water to foam in the boilers and may lead to accidents. Further, though sugar may not be present in sufficient quantity to endanger the boilers through foaming, it is decomposed by the heat into products that are very detrimental to the tubes and shells of the boilers, causing pitting and overheating. The sugar is supposed to be first hydrolized, after which the dextrose and levulose decompose. The dextrose produces levulins, formic and acetic acids, and the levulose, humic and formic acids and insoluble humic compounds. Both the acids and the insoluble humic compounds are injurious to the boiler-plates. Humic compounds form only when the water contains ammonia or soda. Except for its action on levulose in forming humic compounds, ammonia does not appear to injure the plates.

Sugar itself does not attack the boiler metal, but its decomposition products do, both chemically and physically. The chemical action has been explained. Some of the decomposition products deposit upon the heating surfaces with consequent overheating and damage to the plates.

The plates may also be attacked by the acidity in the water derived from sulphited juices. This may be prevented by the addition of soda to the water or preferably by reducing the acidity of the juices.

It is evident that a thorough control of the feed-water is the best safeguard against sugar. The moment sugar appears in the water this should be turned to the sewer and the boilers should be thoroughly blown down. The  $\alpha$ -naphthol test as below should be used at very frequent intervals in testing the water and in tracing the sources of sugar in it. The odor of the steam is very pronounced when the water contains sugar.



**329. Alpha-Naphthol Test for Traces of Sugar in Water.**—This test is widely used to determine the presence of sugar in condenser waters, boiler-feed waters, factory sewer outflows and other waters where the presence of sugar may be detrimental or represent a preventable loss. It is roughly quantitative if carried out under similar conditions and compared with tests on standard solutions. Add 5 drops of a 20 per cent alcoholic solution of  $\alpha$ -naphthol to 2 cc. of water (cooled) in a test-tube, then by means of a pipette reaching to the bottom of the tube add 5 cc. of concentrated sulfuric acid. In the presence of sucrose a violet zone or ring appears at the juncture of the two liquids, the intensity of the color depending on the sucrose present. The acid used must be strictly chemically pure and the  $\alpha$ -naphthol should be of good quality. The solution of this reagent darkens on exposure to light, and should be freshly prepared from time to time.

The test is extremely delicate. When the solution contains as little as one part of sucrose per million parts of water a very pale lilac tint is visible while one part in ten thousand gives almost a black ring due to charring of the sugar by the acid. The colors between these limits range from various shades of lilac through a deep reddish purple. The chemist can best familiarize himself with the color variations by making tests on water to which has been added known amounts of sugar. The colors darken on standing.

**330. Spencer Alpha-Naphthol Apparatus.**<sup>1</sup>—This was devised to expedite the routine tests for sucrose in condenser waters, etc., doing away with the possibility of contamination by contact with the hands or laboratory apparatus. The funnel, Fig. 128, is first washed with repeated portions of plain water, then rinsed with the solution to be tested. After draining it is again filled with the sample and allowed to flow out until only the horizontal portion is filled. Close the three-way cock, add two or three drops of alpha-naphthol solution from the small container, allow to stand a moment, then cautiously open the three-way cock to the acid reservoir and allow several milliliters of acid to back up in the funnel. Note the color reaction as in a test-tube. Then drain off the acid and water and rinse with fresh water.

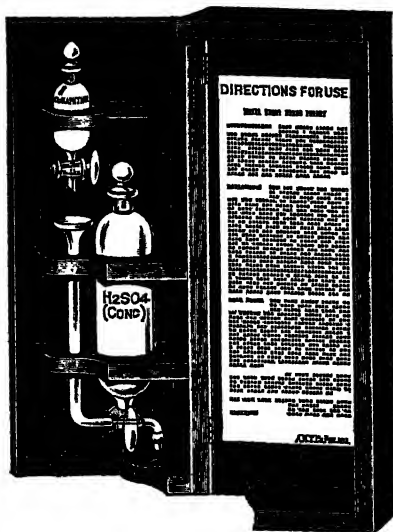


FIG. 128.—Spencer Alpha-Naphthol Apparatus.

<sup>1</sup> Spencer. Ind. Eng. Chem., Vol. 14 (1923), p. 593.

**331. Substitutes for Alpha-naphthol.**—Thymol (U. S. P.) in alcohol may be substituted for alpha-naphthol in the above tests. Lysol (concentrated commercial diluted with 5 parts distilled water) may also be used. A solution of 150 ml. of pure cresol made up to a liter with water containing 60 grams of Castile soap is another reagent that may be used, ten drops to the test. The substitutes are not as sensitive as alpha-naphthol but they are advocated by various authors because the solutions do not deteriorate on standing.

A test using ammonium molybdate was recently described by Matthews.<sup>2</sup>

It is sensitive to one part in 500,000 and offers some advantages through the use of permanent standards for quantitative tests.

Five cc. of the liquid to be tested are placed in a clean test-tube, three drops of concentrated HCl and 3 cc. of a 4 per cent ammonium molybdate solution are added, and the tube is placed in a boiling water-bath for exactly six minutes. If sugar is present a blue color develops, which is, however, not permanent. For quantitative work, standards are prepared by diluting a solution containing 1 gram pure sucrose per liter to convenient concentrations, and treating these solutions exactly as described for the test. Permanent standards may be made by diluting blue-black ink to match the freshly prepared primary standards. For concentrations below 0.0125 per cent of sucrose, dilute Fehling solution must be used instead of ink to get a perfect color match. These secondary standards keep for six months. The conditions of the test must be strictly adhered to in quantitative work, because even slight variations in detail may cause large errors.

**332. Cobaltous Nitrate Test for Sucrose.**<sup>3</sup>—To about 15 cc. of sugar solution add 5 cc. of a 5 per cent solution of cobaltous nitrate. After thoroughly mixing the two solutions, add 2 cc. of a 50 per cent solution of sodium hydrate. Pure sucrose gives by this treatment an amethyst-violet color which is permanent. Pure dextrose gives a turquoise-blue color which soon passes into a light green. When the two sugars are mixed the coloration produced by sucrose is the predominant one, and 1 part sucrose in 9 parts dextrose can be distinguished. If the sucrose be mixed with impurities, such as gum-arabic or dextrin, treat with alcohol or subacetate of lead before applying the test.

**333. Automatic Alarm for Sugar in the Boiler Feed-water.**—This alarm is based upon the change in density of the water in the presence of sugar. It is composed of two communicating tubes (communicating vessels) one within the other. The water is stagnant in the inner tube and flows through the outer at a constant level. A change of level in the inner tube causes a float to rise and close an electrical circuit and ring a bell. Since the water is of the same temperature in both tubes, the density is automatically corrected. The columns are 1.5 meters high, therefore a solution of 1.001 sp. gr. will lift the float 1 mm.

It is evident that the sensibility of the instrument may be increased by adjusting the contact. The instrument should be placed in a convenient location as free as possible of vibrations. A bell should be placed near the feed-water pump and another in the laboratory.

<sup>2</sup> Maryland Acad. Sci. Bull., Vol. 7 (1928), No. 3, p. 35.

<sup>3</sup> Agricultural Analysis, H. W. Wiley, Vol. III, p. 189.

## CHAPTER XXXII

### ANALYSIS OF MOLASSES CATTLE-FOOD<sup>1</sup> (MOLASCUIT)

**334. Notes on Cattle-food Analysis.**—The moisture and the sugar tests are usually required by the factory in the control of the manufacture. The material is usually in excellent condition for the analysis, without further preparation.

**335. Determination of the Moisture.**—Dry a quantity of the food representing about 2 grams of dry material in a tared flat aluminum-dish at the temperature of boiling water under vacuum (100 mm. mercury or less); or dry in a current of dry hydrogen at the same temperature but at atmospheric pressure. Approximately five hours are needed to dry to constant weight:

$$\text{Loss in Weight} \div \text{Weight of Sample} \times 100 = \text{Per cent Moisture.}$$

**336. Determination of the Ash.**—Incinerate 2 grams of the material as described in 252.

**337. Ether Extract.**—Extract 2 to 3 grams of the food, dried as described in Section 335, with anhydrous alcohol-free ether. The extraction is most conveniently made in a percolator, using Knorr's modification of Soxhlet's apparatus shown in Fig. 129. The tube *D* is connected by a cork with a small tared flask containing ether. The outer tube is connected with a reflux condenser and the tube *A* or percolator is prevented from closing the tube *D* by a spiral *C* of copper wire. A syphon-tube *S* is sealed into the lower part of the small percolator *A*, and lies close to the wall. The lower part of the tube *B* is closed with a perforated disk of platinum.

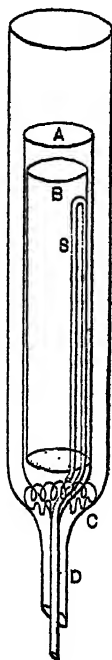


FIG. 129.  
Knorr's Extraction Apparatus.

In using the apparatus the weighed material is placed in *B*, with a light plug of ether-extracted cotton on top of it, and the percolator is connected with the reflux condenser and the ether-flask; this latter is placed on a water-bath and the water is heated with an electric lamp or by steam. In using a flame there is danger of igniting the ether. The ether vapors ascend to the condenser where they are condensed and the ether drips into the tube *B*. The tube *B* fills with ether to the top of the syphon and is then carried back by this into the flask. This extraction should be continued for sixteen hours. The ether is removed from

<sup>1</sup> Partly based upon methods of Official Association of Agricultural Chemists.

the flask by evaporation and the extract is dried in a water-oven to constant weight. The percentage that the residue is of the original material is reported as ether extract. If preferred the extract may be indirectly ascertained by determining the loss in weight of the material in the tube *B*.

**338. Crude Protein.**—Determine the nitrogen by the Kjeldahl method as described in any work on quantitative analysis and multiply the percentage by 6.25 to ascertain the per cent of crude protein.

**339. Crude Fiber.**—Extract 2 grams of the food with ether and to the residue, in a 500-ml. flask, add 200 ml. boiling 1.25 per cent sulphuric acid. Connect the flask with a reflux condenser and boil the liquid for exactly thirty minutes. A blast of air conducted into the flask may serve to reduce the frothing of the liquid. Filter and wash the residue with boiling water until the washings are no longer acid. Rinse the material back into the same flask with 200 ml. of boiling 1.25 per cent solution of sodium hydroxide and boil for thirty minutes as before. The sodium hydroxide should be free or nearly so from sodium carbonate. Collect the residue in a tared Gooch crucible and wash it with boiling water until the washings are neutral. Dry the residue at 110° C. and weigh it; incinerate it completely and again weigh. The loss of weight is calculated as crude fiber.

The filter used in the first filtration may be of linen, asbestos, or glass wool. The acid and alkali solutions should be standardized by titration.

**340. Sucrose and Glucose.**—These are determined by chemical methods. The solution for analysis is prepared by extracting 10 grams of the material with 125 ml. of 50 per cent alcohol (by volume) adding about 3 grams of calcium carbonate to neutralize acidity and boiling for one hour in a 250 ml. flask. A small funnel is placed in the neck of the flask to condense the vapors. Cool and allow to stand overnight. Make to the mark with neutral alcohol, 95 per cent, mix and allow the insoluble matter to settle. Drain off 200 ml. of the clear liquid, place in a beaker and evaporate on a steam bath to a volume of about 25 ml., then rinse into a 100-ml. flask with water, add sufficient neutral lead acetate solution to clarify, make to the mark, mix and filter. Delead with dry sodium oxalate and filter again.

Determine reducing sugars by the Munson-Walker method using 25 ml. (2 gms. material) of the delead solution diluted to 50 ml.

For the sucrose determination invert 50 ml. of the delead solution by the method given in 239A making to 100 ml. after neutralization. The invert sugar is again determined on 50 ml. of the inverted solution (2 grams original material) by the Munson-Walker method and the sucrose calculated as described in 239A.

To correct for the volume of insoluble material in the food, multiply the percentages of invert sugar and of sucrose by 0.97, this figure having been arrived at by experience.

## CHAPTER XXXIII

### DEFINITIONS AND APPLICATIONS OF EXPRESSIONS USED IN SUGAR WORK

**341. Normal Juice. Undiluted Juice. Absolute Juice.**—The normal or undiluted juice was originally assumed to be the juice as it actually exists in the cane. These expressions are now applied to the juice extracted by dry-milling, *i.e.*, milling without saturation of the bagasse. The expression “undiluted juice” is perhaps preferable to “normal juice,” but long usage has established the latter.

The cane is known to contain water that is free of sugar (279). This is termed “colloidal water.” If a piece of cane be passed between the rolls of a mill a part of this water exudes and drips from the end of the stalk. In view of the presence of this water it becomes difficult to define the juice as it exists in the cane, in the light of factory requirements. For calculations based upon the whole or normal juice of the cane, it may be well to consider this the water-soluble constituents—juice solids dissolved in all the water contained in the cane. This may be termed the “absolute juice.” This would assume that all the cells may be broken down and the solids be distributed in their liquid content. The cells are never all ruptured in milling, therefore the juice extracted in dry milling can only approximate the “absolute” juice. This fact has an important bearing upon the inferential methods of calculating the weight of the cane, saturation-water, juice content, etc. The customary use of “normal juice” as explained above is that employed in this book. The analysis of the normal juice is calculated from the density of the crusher or mixed-crusher and first-mill juice, and the purity of the mixed or diluted juice (269). A factor is calculated from the density of the crusher-juice and that of the mixed juices obtained in dry-milling (*i.e.* when no saturation water is used.) This factor is applied to the crusher-juice density to ascertain that of the normal juice. Example. Crusher-juice Brix, 20°; mixed juice Brix, 19.7; factor or ratio =  $19.7 \div 20 = 0.985$ . See also page 369.

**342. First Expressed Juice.**—This is undiluted juice from the crusher, or crusher and first mill, or in those factories having no crusher, from the first mill alone. The per cent sucrose is used in the calculation of the Java ratio and the degree Brix in the calculation of the analysis of the normal juice.

**343. Mixed Juice. Dilute Juice.**—These expressions are used to indicate the juice extracted by all the mills as it is finally mixed and sent to the defection-station. This juice is usually diluted with the saturation water.

When no saturation water is used the "mixed juice" and "normal juice" are identical.

**344. Bagasse. Megasse.**—This is the woody residue left after expressing the juice from the cane. The word "megasse" is used in the English colonies.

**345. Residual Juice.**—The bagasse may be regarded as a sponge that absorbs and retains a part of the juice. The juice so retained, the residue of that in the cane, is the "residual" juice. The true residual juice can only be approximated and in the analysis only the coefficient of purity of an assumed residual juice is determined. This number is used in calculating the percentage of fiber or marc in the bagasse and cane. In practice the juice flowing from the last mill of the train or the last roll of that mill is considered to have the same coefficient of purity as the true residual juice. The juice from the discharge-roll of the last mill is recommended for this test.

**346. Fiber or Marc.**—This is the water-insoluble matter of the cane. The true fiber or cellulose is not determined in the factory control.

**347. Extraction. Mill Extraction. Dilute Extraction.**—This is the percentage of dilute or mixed juice on the cane.

*Normal Extraction.*—Similarly, this is the normal juice per cent cane.

*Sucrose Extraction.*—The sucrose in the mixed juice in terms of percentage of sucrose in the cane.

A discussion of these various extraction figures and their value will be found in 378.

**348. Saturation Water. Saturation. (Maceration. Imbibition).**—The water which is sprayed on the partly ground bagasse as it passes from one mill to the next is termed saturation, maceration or imbibition water, the terms now being generally used synonymously. The "saturation" (maceration, imbibition) is the amount of this water expressed in percentage terms of the cane.

**349. Dilution.**—That portion of the saturation water which is extracted with the juice is termed the dilution water. "Dilution" is the percentage of this water in terms of the weight of cane. It is calculated from the respective degrees Brix of the normal and dilute juices.

**350. Java Ratio.**—This arbitrary milling ratio, first used in Java, is the relationship between sucrose in the "first expressed juice" (generally the crusher juice) and the sucrose in the cane.

$$\text{Java Ratio} = \frac{\text{Per Cent Sucrose in Cane}}{\text{Per Cent Sucrose in First Expressed Juice}} \times 100.$$

This relationship has been found to be fairly constant for any given mill equipment irrespective of normal variations in the fiber content of the cane. If it is calculated for any given sugar factory over a sufficiently long period it becomes of value as an indication of the accuracy of cane weights and juice weights, as well as the sampling and analysis of the juices and bagasses. It does not afford any comparison between the work of one factory and another

and a radical change in milling equipment may alter the ratio quite appreciably for any given factory. (See also 371.)

The Java ratio has been used in Natal to figure the sucrose in the cane, as delivered by the grower, from the sucrose in the crusher juice of that cane.<sup>1</sup>

**351. Sucrose Per Cent Fiber.**—This is the relationship between the sucrose in the bagasse in percentage terms of the fiber in the bagasse. Since it takes into account the inert fiber which serves as a vehicle for the sucrose lost in bagasse it is considered a more valuable measure of milling efficiency than the per cent sucrose in bagasse alone.

**352. Defecated Juice. Clarified Juice.**—The dilute juice after it has passed through the clarification system.

**353. Sirup.**—The sirup is the concentrated juice of the cane from which no sugar has been extracted. This is the "meladura" of the Spanish-American factories. The word "sirup" has an opposite meaning in sugar refineries where it is applied to solutions from which sugar has been removed by crystallization.

**354. Massequite.**—The massequite is the concentrated sirup or molasses in which the sugar has been crystallized or the material has been concentrated to a point where it will crystallize. Massequites are designated by names, numbers or letters indicating their purity or the number of crops of crystals of sugar that are to be removed.

**355. Molasses.**—When a massequite is spun in a centrifugal machine the sugar crystals are separated from the mother liquor. This liquid is now termed "molasses," and is designated by names, numbers or letters corresponding with the massequites. The "final" or true molasses is the liquid residue from which no more sugar can be removed, either on account of factory equipment or for commercial reasons. This is termed "barrel-sirup" in the refineries.

**356. Circulating Water. Condenser Water.**—This is the water used in condensing the vapors in the evaporation of the juice and sirup. After leaving the condensers, this water (in the tropics where water is scarce), together with that derived from the vapors, is usually passed over a cooling-tower to reduce its temperature, and it is returned to the condenser, thus circulating through this apparatus. This water is often termed "cooling-tower water."

**357. Sweet-water.**—With the older types of apparatus, the vapors condensed in the calandrias of the evaporator often contained sugar, carried into it by entrainment. The water resulting from these vapors was called "sweet-water," and still receives this name, though with modern apparatus it contains no sugar. In refinery practice any very dilute sugar solution that is returned to the process is termed "sweet-water."

**358. Entrainment.**—When sugar is carried off with the vapors from the evaporators and vacuum-pans, this is called "entrainment."

**359. Coefficient of Purity.**—The coefficient of purity as usually applied is the percentage of apparent sucrose in the apparent solids (Brix) of the material. This coefficient is calculated by dividing the per cent sucrose, as

<sup>1</sup> Bechard. So. African Sugar Jour. Vol. 12 (1928), p. 177.

ascertained by direct polarization, by the degree Brix of the substance and multiplying the quotient by 100. The number calculated in this way is not the true coefficient of purity but only the *apparent coefficient*. The *true coefficient* of purity is calculated in the same manner, except that the percentage of solid matter as ascertained by actually drying the material and the sucrose as determined by the Clerget or double-polarization method are used.

The usage in the sugar industry is to apply the term "coefficient of purity" (or more generally simply "the purity") to the number when calculated from the apparent degree Brix and the sucrose by direct polarization. It is well known that this coefficient so calculated is an approximate number, but for comparative purposes it is of great value in sugar manufacture and will doubtless always be so used. Frequently approximate data of the purity of a product are required for immediate use, and as the factory superintendent cannot wait for a tedious determination of solids and an even more tedious double polarization, he uses the apparent purity, bearing in mind its shortcomings.

Since allowances must be made, it is advisable that the analyses always be conducted under very similar conditions, that due weight may be given the purity.

On the other hand, the *true coefficient of purity* is only used in special researches and in making comparisons among the various products at different stages of the manufacture or in comparing the work of two or more factories. In using this coefficient the word "*true*" should never be omitted as is often the case, rendering what would otherwise be valuable data almost useless.

The expressions "quotient of purity," "degree of purity," "exponent of purity," and often "the quotient," and the "exponent" are used in referring to this coefficient. The commonest expression used is simply "the purity."

With modern methods of sugar-house work, "boiling in" molasses and using crystallization in motion, the apparent purity is very frequently required and is of very great value. (See page 326.)

**360. Sucrose. Polarization.**—These terms are frequently used synonymously in cane-factory work to designate the direct polarization. The Clerget polarization is called "true sucrose" or the "Clerget" to differentiate it from the direct polarization figure. For brevity and simplicity this practice is followed throughout this book.

**361. Glucose Coefficient, Glucose per 100 Sucrose, Glucose Ratio.**—This number is calculated as follows:

$$\frac{\text{Per cent glucose} \times 100}{\text{Per cent sucrose}} = \text{glucose ratio.}$$

This coefficient is useful in detecting inversion of sucrose in the manufacture. Provided no sucrose has been separated from the material and no glucose has been removed or destroyed, an increase in the glucose coefficient indicates inversion.



It is possible that by the destruction of both sucrose and glucose the relations between the two sugars might remain the same and yet inversion have occurred.

**362. Saline Coefficient. Ash Ratio.**—The saline coefficient is the quantity of sucrose per unit of ash.

*Calculation:*

$$\frac{\text{Per cent sucrose}}{\text{Per cent ash}} = \text{ash ratio.}$$

**363. Molasses Factor.**—This is the ratio between the sucrose lost in molasses and the non-sucrose entering the house in the juice.

$$\frac{\text{Suc. in Mol. \% Cane}}{(\text{Bx. Normal J.} - \text{Sucr. Normal J.}) \times \text{Norm. J. \% Cane}} = \text{Molasses Factor.}$$

This is a valuable figure in comparing the molasses work of different factories since it takes into account the percentage of molasses made and the sucrose content of that molasses, in relation to the amount of molasses-making material (non-sugar) entering the factory. It is quite evident that all these factors must be considered for a true comparison. The molasses factor fails to take into account the impurities going into the raw sugar. For this reason, the factor gives strictly comparable figures only where raws of the same test are turned out. Obviously a factory making raws of 97° test will make more molasses than one turning out 96° test raws (all other conditions being equal).

**364. Available Sugar.**—Several formulæ are in use for the calculation of available sugar or the sugar it is assumed that a factory should be able to obtain with juices of a given analysis. Manifestly there are several conditions that control the proportion of sugar that may be considered available in the mill-juice, viz.: the efficiency of the machinery, the quality of the juice, and the skill of the factory superintendent. In considering the quality of the juice, the nature of its impurities, as well as its richness, should be taken into account. A cane grown on certain soils may take up more melassigenic substances than one of the same richness and equal apparent purity from another soil, and consequently the proportion of actually available sugar would be quite different, whereas a formula would indicate the canes to be equal.

Available sugar-numbers are but rough approximations except when the elements of the factory equipment and efficiency are taken into consideration. The yield or recovery of sugar varies with the coefficient of purity of the juice and the losses in manufacture, hence a formula for available sugar should take these elements into account.

The practical application of available sugar calculations except in estimating return from new investment, is in comparing the present work of a factory with that of some previous period or with that of a factory similarly located as regards soil and climate. Such calculations are of value in the control of several establishments operated under a central organization. In this event it is desirable to know (1) without stock-taking, how nearly a factory is approaching its previously demonstrated efficiency; (2) how much sugar is in process to determine whether the factory is becoming congested and requires either a

reduced grinding-rate, a modification of the process, or closer supervision of the personnel; (3) how much sugar is in process for commercial or accounting reasons and (4) whether it is more profitable to reduce the rate of grinding or sacrifice somewhat in juice extraction.

Available sugar estimates also become necessary when cane is purchased on a basis of its analysis.

The earlier formulæ for available sugar were evidently based upon refining experience and possibly that of beet-sugar factories. The conditions in both of these branches of the industry are very unlike those of the cane-sugar factory and will not apply very fully in the calculation of the production of sugar from cane.

*Formula of Winter and Carp.*—This formula, published by Prinsen-Geerligs,<sup>2</sup> is based upon experience in Java but it has found wide use throughout the sugar world. Many modifications have been suggested but the original formula is still general:

$$x = S \times \left( 1.4 - \frac{40}{C} \right),$$

where

$x$  = Available sucrose per cent cane;

$S$  = Per cent sucrose in the juice in terms of the weight of the cane;

$C$  = Coefficient of purity of the juice.

To calculate the available sucrose to terms of available sugar divide the value  $x$  by the polarization of the sugar and multiply by 100.

The derivation of the formula is based on Winter's observations of actual results in Java which showed that 1 part of non-sucrose held 0.4 part of sucrose in the final molasses. If  $C$  is the purity of the juice and  $S$  the sucrose in the juice per cent cane (as above) then:

From the Brix-Purity-Sucrose relationship

$$100S/B = C \text{ or } B \text{ (Solids in Juice Per Cent Cane)} = 100S/C$$

$$\text{Non-sucrose in Juice Per Cent Cane} = 100S/C - S.$$

Therefore from the conditions assumed above that .4 pound of sucrose go to molasses for each pound of solids.

$$\text{Available Sucrose Per Cent Cane} = S - .4(100S/C - S) = S(1.4 - 40/C)$$

*Note.*—It will be seen that if 0.4 part of sucrose occurs in the molasses for every part of non-sucrose the total solids must be 1.4 parts and the purity of the molasses is  $.4/1.4 = 28.57$  purity. This is either "true" or "apparent" purity, depending on whether "true" or "apparent" sucrose and solids are used in the calculations.

If this molasses purity of 28.57 is substituted in the available sucrose formula for purities (p. 384);

$100(C - M)/C(100 - M)$  where  $C$  = the juice purity, and  $M$  the molasses purity, the result will check that part of the Winter-Carp formula  $(100(1.4 -$

40/C)) which gives the theoretical retention calculated for the various juice purities in the table on p. 487. Taking the first figure in the table as an example

$$100(77 - 28.57)/77(100 - 28.57) = 88.05.$$

It should be noted that the Winter-Carp formula takes no account of the losses in manufacture.

The formula of Winter-Carp-Geerligs was put to many uses by Dr. Spencer. When used in connection with an efficiency number (366) the yield or recovery of sugar may be estimated with very considerable accuracy from the weight of the cane, the mill extraction and the analysis of the juice. It is perhaps needless to state that the analytical data must be based upon accurate sampling. Should the sampling be inaccurate these calculations will call attention to the inaccuracy.

The second part of the formula,  $100(1.4 - 40/C)$  gives the "sucrose retention" or "recovery" number (365), and this number divided by 0.96 gives the recovery in 96° sugar. A table of the values of  $100(1.4 - 40/C)$ , and this value divided by 0.96 is given on pages 487, 488.

The following example illustrates the use of this formula and the efficiency number (366), in calculating the available 96° sugar:

Sucrose in the cane, per cent.....	14.1
Extraction, sucrose per cent sucrose in the cane.....	93.49
Coefficient of purity of the juice.....	84.1
Boiling-house efficiency number.....	99

Sucrose extracted per cent cane =  $14.1 \times .9349 = 13.18$ ; referring to the second part of the table, page 488, the recovery number corresponding to 84.1 coefficient of purity is 96.29 and  $13.18 \times .9629 = 12.69$ , the available 96° sugar per cent cane if the efficiency number were 100. Applying the actual efficiency number, 99, we have  $12.69 \times .99 = 12.56$ , the available 96° sugar.

The following "run" and "to date" yields of a large Cuban factory indicate the reliability of this method for available sugar:

#### 96° CENTRIFUGAL SUGAR PER CENT CANE

For the Run		To Date	
Actual Yield	Calculated	Actual Yield	Calculated
9.96	10.13		
10.72	10.58	10.35	10.28
11.50	11.38	10.70	10.68
11.85	11.84	10.98	10.98
12.44	12.33	11.31	11.39

It should be remembered that "run" figures are more liable to fluctuations than "to date," as they may be affected by the errors of estimate of two runs. It should be stated that this factory was operated beyond its normal capacity, thus necessitating slight changes in its efficiency number from time to time. This affects the estimate of the available sugar as the "to date" efficiency number, for the previous run is used in the calculation. Also in the first run the average number for the previous crop is used.

**365. Sucrose Retention or Recovery.**—This number is the percentage of the sucrose in the extracted juice that is retained or recovered in the commercial sugar. In the following example of a sucrose balance, 92.32 is the retention number:

Sucrose in the extracted juice, per cent.....	100
Sucrose in the sugar per cent sucrose in the extracted juice.	92.32
Sucrose in the molasses per cent sucrose in the extracted juice.....	6.58
Sucrose in the press-cake per cent sucrose in the extracted juice.....	.44
Sucrose in the undetermined per cent sucrose in the extracted juice.....	.66 100

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**366. Boiling-House Efficiency Number (BHE).**—Many factories use efficiency numbers to show how nearly a theoretical yield of sugar is obtained. This number is usually the percentage relation between the actual retention or recovery number (365) and the number based upon an available sugar formula. A part of the Winter-Carp-Geerligs formula,  $100(1.4 - 40/C)$ , was used by Spencer in calculating the Efficiency Number of The Cuban-American Sugar Co.'s factories. This calculation is best illustrated by an example: Let the coefficient of purity of the raw juice be 86.0 and actual retention number be 92.32, as in the previous paragraph, then  $100(1.4 - 40/86) = 93.49$ ;  $92.32 \div 93.49 \times 100 = 98.7$ , the efficiency number. A table is given on page 487 from which the value of  $100(1.4 - 40/Purity)$  may be ascertained by inspection.

The efficiency number is of value in calculating sugar actually available, as it takes into account the losses in manufacture in the particular factory as compared with a factory operating under the assumed conditions of the available sugar formula. This number may, in rare cases, exceed 100, but generally this occurrence should cast doubt on weights, sampling or analytical work.

## CHAPTER XXXIV

### CHEMICAL CONTROL OF SUGAR-HOUSE WORK

**367. Introductory**—The chemical control of the factory is intended primarily to guide the manufacture along lines of best practice and to assist in detecting and reducing losses of sugar.

The control of the milling considers the cane from its delivery to the crusher to the delivery of the juice to the boiling-house and the bagasse to the fires. It is quite as important to ascertain the loss in the bagasse as to report the extraction of juice and sucrose to the engineer. The engineer should not so much consider the proportion of sugar extracted as that lost. The effort should always be the reduction of losses. It requires the assistance of the chemist in locating the causes of these losses.

The crystallization of the sugar should receive constant attention. The purity coefficients of the massecuites should be maintained at certain numbers, that the pan-work may progress systematically and efficiently and that no unnecessary work be thrown upon the crystallizers. This facilitates the reservation of a large part of the crystallizer capacity for the low-purity massecuites. The crystallizers must be controlled to meet the best conditions of equipment and manufacture. Molasses is sometimes sold on a basis of its test, and in this event the control of the massecuites becomes of additional importance.

The quality of the sugar must be controlled to maintain it upon a basis of the most profitable analysis. The moisture content must be kept within certain limits to protect the sugar from deterioration in storage.

**368. Duties of the Chemist.**—The chemist in the factory corresponds with the auditor in the accounting department. He charges the superintendent with the sucrose entering the factory in the raw material and credits him with that leaving it in the products, by-products, and losses. It is his duty to trace the travel of the sucrose and locate losses.

The chemist is also the statistician of the factory. He reports the quantity of the raw materials, the analysis of the materials in process of manufacture and of the products and by-products. The chemist prepares statistics that have a bearing upon the control and economy of the manufacture and the business of the establishment.

Research work in connection with the improvements in the equipment or processes is often called for and this often necessitates both technical and chemical training on the part of the chemist.

The laboratory should be a part of the training-school for future superintendents. The chemist becomes acquainted with all the methods, problems

and difficulties of the manufacture. His control of the work should familiarize him with the details of the processes. The chemist's training unfortunately lacks the opportunity for practice in directing and controlling labor. This must come to him before he can leave the school of the superintendent. He should also acquire a good working knowledge of pan-boiling.

A lesson that must be learned early in factory control is that samples must be representative of the materials and their integrity must be unquestionable. Apparatus and methods must be adapted to the work in hand. Where the highest feasible accuracy is required, as in the testing of the raw materials and the products, no detail should be omitted or labor spared that may lead to dependable results.

**369. Outline of Factory Control.**—The following is an outline of the factory control. This may be greatly extended by introducing "true" numbers instead of apparent for sucrose, etc., but this does not usually appear necessary in routine control.

The weight of the cane is reported to the chemist and from this number and the weight and analysis of the raw juice extracted he calculates the mill-control numbers for the engineer and the weight of sucrose chargeable to the superintendent. He must later account for this sucrose in the products, by-products and losses. The mill control depends very largely upon the analysis of the bagasse, and in some cases, entirely upon this analysis.

The raw juice is the starting-point in the control of the manufacture. Its weight and analysis are also elements of the mill control. The weight of the sucrose extracted in the juice plus that remaining in the bagasse is the basis of the determination of the percentage and weight of sucrose entering in the cane and for the calculation of the sucrose extraction.

The analysis of the sirup or concentrated cane-juice, the "meladura" of the Spanish-American factories, is the control of the purification of the juice and its evaporation, and is a guide in the sugar-boiling. To obtain satisfactory results in pan-boiling and to bring this work to as nearly a scientific basis as is possible, the analysis of the massecuites and molasses is necessary. Massecuites are now usually boiled to specified purities by the injection of molasses. Careful control of this work is essential to a systematic grading of the materials for the most profitable extraction of the sugar, considering the limitations of the factory. Frequent control tests are also required in the conduct of the crystallizers. The products, sugar and molasses, are controlled both as a check upon the manufacture and to meet market conditions.

The filter-press cake is one by-product whose weight may be readily ascertained. Its analysis is usually limited to the determination of the sucrose, though occasionally the solids must be determined for controlling the efficiency of the pressing and the quantity of water used in "sweetening off," or in reducing the cake for refiltration.

A loss of sucrose may occur through entrainment in the multiple-effects and vacuum-pans. A knowledge of the temperatures of the condensing and condenser-waters is required in estimating this loss. (See p. 442.)

The chemist must judge from the equipment of the factory what control work may be omitted without decreasing the efficiency of the sugar-house.

He must under all conditions determine when he may properly, to some degree, sacrifice accuracy for the sake of promptly obtaining approximate figures for immediate use.

It is just as important, that the necessary chemical control be complete and the laboratory records well arranged as that the accounts of the various departments be full and accurate.

#### WEIGHTS AND MEASURES

**370. General Considerations in Regard to Weights and Measures.**—It is quite essential that all weights and measures should be of the same system or be reduced to the same system. This remark applies especially to Spanish America, where it is not unusual to find English and Spanish units used indiscriminately.

The system of weights and measures adopted should be applied as accurately as possible. A checking-system should be devised to meet the conditions of the factory and in so far as may be possible eliminate errors in the calculations.

**371. Cane Weights.**—There are few factories that do not weigh their cane. Cane weights, however, are not essential to the control of the boiling-house, and in a measure may be dispensed with in mill control. In this event the mill control depends entirely upon the analysis of the bagasses and the juices.

It is the general custom to use the Spanish pound in weighing cane in Cuba. These weights are reported in arrobas of 25 pounds, Sp. The ton (*tonelada*) contains 2000 pounds (*libras*) Sp. There is some confusion on the part of scalemakers as to the equivalence of the Spanish pound, though the leading scales are properly graduated. A decree of a Spanish Captain General in Havana gave the equivalence as 100 Spanish pounds = 46.0096 kg., therefore 100 Spanish pounds = 101.4338 avoirdupois pounds. Scale-beams graduated in Spanish pounds use the word "libra."

It is the custom in Cuba and in most factories to reweigh the cane on a central scale immediately before grinding it. This weight should be used in the control.

It may occur through conditions beyond the control of the chemist that the weight of the cane must be estimated by an inferential method. Small factories, and even the large ones of Louisiana, often store the cane in large piles, liquidating these as often as is practicable. In certain localities the cane is flumed or floated to the mills in water. These special conditions call for an inferential method. Such methods should only be used in estimating the cane for statistical purposes and not in the serious control of the factory. Inferential methods require a knowledge of some constituent of the cane that may be traced through the milling process, *e.g.*, the fiber, solids, or sucrose. The following example, from the records of a factory, illustrates an inferential method:

Use the Java ratio (350) which gives the relationship between the sucrose in the crusher and the sucrose in the cane. This factor varies in Cuba from about 84 down to 77, the average being close to 81 for all the factories in the

Cuba Sugar Club; the heavier the crushing the higher the ratio, of course. Figures as high as 90 or above have been reported by Hawaiian mills using shredders.<sup>1</sup>

Deerr<sup>2</sup> found factors varying from a minimum of 0.81 to a maximum of 0.848 and an average of 0.825. According to Pellet<sup>3</sup> the factor in Egypt is usually from 0.83 to 0.84 and may be as low as 0.82 to 0.80.

Java ratio.....	0.80
Polarization of the crusher juice.....	18.45
Tons sucrose extracted in the mixed juice.....	305.7
Fiber in the cane (direct test).....	11.3
Fiber in the bagasse (direct test).....	48.9
Bagasse per cent cane = $11.3 \times 100 \div 48.9 =$ .....	23.11
Sucrose in the bagasse, per cent.....	4.5
Sucrose in the cane, per cent = $18.45 \times 0.80 =$ .....	14.76
Sucrose in the bagasse per cent cane = $23.11 \times 0.045 =$ ...	1.04

Weight of cane = sucrose extracted  $\div$  sucrose per cent in cane - loss in bagasse =  $305.7 \div (14.76 - 1.04) = 2228$  tons. The weight reported from the central scales was 2209 tons, showing the factor to be approximately 0.80. In this example, taken from the records of a well-controlled factory, the fiber in the cane was actually determined by the indirect method. Where an inferential method must be used a direct fiber determination must be made, thus adding to the uncertainty of the method. In this method, but three elements are accurately determined, viz., the polarization of the crusher juice, the sucrose extracted by the mills and the analysis of the bagasse.

**372. Weight of the Juice.**—Practically all factories of any size whatever in Cuba and Porto Rico weigh the mixed juice in tank scales, hand operated. These scales are tanks with conical bottoms to facilitate washing, arranged in pairs so that one is emptying while the other is filling. Counters, printing counterpoises and other control devices are used with these scales. A factory grinding about 2000 tons of cane per day requires two tanks, each holding about five tons of juice.

On the accuracy of the weight of the mixed juice depends the entire control of the cane factory and extreme care should be taken at this station to avoid even small errors, particularly those which may tend to become cumulative.

Automatic juice scales are on the market but are not in use in sufficient numbers to warrant comment. The simplicity of the hand-operated scales gives them the advantage over automatic devices.

**373. Measurement of the Juice and Calculation of Its Weight.**—The measurement may be made with a fair degree of accuracy in factories using separate liming-tanks though the results are never as satisfactory as with juice-scales. At least three tanks are required and these should be connected with a much deeper tank, serving as a pump-tank. The pump-tank must be deep enough to admit of complete drainage of the liming-tanks. The tank-valves

<sup>1</sup> The Planter. Vol. 68 (1922), No. 2.

<sup>2</sup> Int. Sugar. Journ., 1911, 13, 15.

<sup>3</sup> Int. Sugar. Journ., 1912, 14, 587.



should be well ground to prevent juice from passing from a full to an empty tank. All of the tanks should have conical bottoms and spray-pipes for washing. An overflow-pipe should connect each tank and with a trunk line leading back to the mills. A "T," inside the tank, should connect by the side branch with the overflow-pipe. The upper outlet of the "T" should be extended well above the foam on the surface of the juice and the lower outlet a few inches below the surface, by nipples. The object of this arrangement is the provision of a free overflow for the juice, without interference by foam. A 3-inch "T" is a suitable size.

The measuring-tanks should be calibrated with water under service conditions. Corrections must be applied to the measured volume for temperature, milk of lime added and for air entrained with the juice. The allowance for air should be determined experimentally. It varies with milling and pumping conditions. A tank should be filled to the overflow with juice and its temperature be noted. After a few hours the temperature and shrinkage of the juice should be noted. A factor should then be figured from these data, making allowance for change due to temperature. It is advisable to add formaldehyde to the juice to insure its preservation and to make the period of rest as long as is practicable. Occasionally factories warm the juice moderately on its way to the liming-tanks. This reduces the error due to entrainment of air. The method of calculating the weight of the juice from its volume is given on page 386.

Some small factories are compelled to measure the juice in the defecators. Such measurement is very unsatisfactory. A block of wood should be arranged to indicate the point to which the vessel is to be filled. Correction must be made for the milk of lime used and for temperature. It is customary to heat the juice the moment the heating surface is covered, reaching the "cracking" temperature about the moment the defecator is full. This expels the air and gases which carry a part of the precipitated impurities to the surface with them. This scum adds to the difficulty of making an accurate measurement. The calculations are made as given on page 386. Apparatus for the automatic or semi-automatic measuring of the juice eliminate some of the sources of error in hand-measurement but these devices are little used and not to be recommended.

**374. Measurement and Weight of the Sirup.**—The weight of the sirup is not usually required, except in taking account of the stock of the material in process.

It is the present tendency of the factories, in view of improvements that have been made in the defecation, to hold the sirup only long enough to meet the pan requirements.

The sirup must be occasionally measured in taking account of the sugar in process for the run reports. For this purpose the tanks should be gauged and the volume per inch of depth be tabulated. In these measurements it is more convenient to note the "inches out" and figure the sirup in the tank by difference.

**375. Measurement and Weight of the Masseccutes.**—The measurements in the mixer and especially in the crystallizers should be made immediately on

striking the massecuite, on account of its increase in volume after stirring or further crystallization.

The measurement or weight of the massecuite is usually only required at the end of a "run" or period for calculating the quantity of sugar in process of manufacture. In very large factories the errors in these measurements, when carefully made, are so small as compared with the amount of material in process that they may be neglected.

A sample of the massecuites should be drawn, when striking, for analyses for use in making the necessary calculations and for the guidance of the sugar-maker in the conduct of the pan-work.

**376. Sugar Weights.**—The accurate weighing of the raw sugar is of equal importance with the juice and cane weights and this phase of the technical control should be under constant supervision both by the laboratory and the factory superintendent.

Dependable automatic scales, of a type similar to those that have long been used in the weighing of refined sugar, are now generally employed for weighing raws. These use a mechanical arrangement to regulate the flow of the sugar into the weighing hopper and to adjust the "dribble" or last small quantity. One type of these scales will accurately weigh more than 100 bags of 325 pounds of raw sugar per hour. Because of the sticky nature of raw sugar the scales should be frequently cleaned and the delivered weight checked. Richardsons' and the "Libra" Scales are in general use in Cuba.

**377. Measurement of the Molasses.**—The measurement of the molasses is accompanied by difficulties arising from the nature of the material itself. Molasses is very viscous and drains slowly from the containers, making successive measurement in a tank uncertain. Further, heavy molasses occludes considerable quantities of air. This occlusion of air raises the question of a definition of the commercial gallon of final molasses. (See 298.)

Molasses that has been reduced with water preparatory to reboiling is readily measured in the tanks. Final molasses should be weighed in tank-cars when possible, on account of its importance in the factory control. A "wantage table" is given on page 425 for the measurement of the molasses in horizontal cylindrical tanks (tank-cars). The amount of air occluded varies with the container, the method of filling it and the period that elapses between filling and measuring. As has been stated (298) a molasses may weigh less than 11 pounds per U. S. gallon, measured immediately after filling a tank-car while the air-free weight of the same molasses was 12 pounds. This indicates the importance of experimental data as a basis of calculation of the weight of the molasses in meeting local conditions. A float measurement, using a copper float, may be employed for molasses stored in very large tanks.

**"Pneumercator" Attachment for Molasses Tanks.**—The "Pneumercator" is a patented device for weighing the material in a tank by means of the pressure on a delicately adjusted air chamber communicating with a mercury gauge calibrated for each particular tank. The gauge is marked to register in any convenient unit; pounds or tons being the usual markings. The hydrostatic pressure of the molasses on the air-chamber is the actuating force, so all question of foam and allowance for occluded air is eliminated. Many

checks on this device have been made against actual weighings of Cuban molasses and the agreement is highly satisfactory. The pneumericator has eliminated all the difficulties enumerated above concerning the measurement of molasses.

## MILL EXTRACTION

**378. Mill Extraction.**—The quantity of juice or sugar removed from the cane by the mills, the mill extraction, is usually expressed in two ways, viz., (1) (*Juice Extraction.*) The weight percentage on the cane of the mixed juice calculated to terms of the density of the normal juice. This expression is gradually being superseded by the second, which has become quite reliable through improvements in analytical and milling methods. (2) (*Sucrose Extraction.*) The weight of sucrose extracted in the juice per cent of the weight of sucrose in the cane. This number owes its almost universal adoption not only to the fact that it directly indicates the sucrose extraction, but also because it is less influenced by the variations in the composition of the cane. This number was formerly unreliable because it was based upon the direct analysis of the cane or upon a sucrose number derived in the incorrect assumption that the juice as it exists in the cane is of uniform composition.

(1) *Extraction in Weight Terms of the Cane and Normal Juice.*—Divide the weight of diluted or mixed juice (372) by the weight of the cane and point off for percentage, to ascertain the per cent dilute extraction; calculate the dilution per cent cane (390) and subtract this number from the dilute extraction number. The remainder is the extraction of normal juice per cent cane.

(2) *Extraction in Percentage Terms of the Weight of Sucrose in the Cane and the Extracted Juice.*—Calculate the weights of sucrose in the diluted juice and in the bagasse. The weight of the bagasse is ascertained by subtracting the weight of the diluted juice from the sum of the weights of the cane and saturation-water. The sum of the weights of sucrose in the extracted juice and the bagasse is the weight of sucrose in the cane. The weight of sucrose in the extracted juice divided by that in the cane and the quotient multiplied by 100 is the extraction number in terms of the sucrose in the cane and in the extracted juice.

(3) *Extraction in Dry Milling, i.e., without Saturation.*—The calculations are similar to those of (1) and (2) except that no water of dilution need be considered.

Example illustrating methods of calculating the extraction:

Tons of cane ground .....	3254
Ton of dilute juice extracted .....	3450
Tons of saturation-water applied to the bagasse .....	877
Degree Brix of the crusher juice .....	19.21
Factor for reducing the Brix of the crusher juice to that of the normal juice, (341) .....	0.97
Degree Brix of the diluted juice .....	14.52
Per cent sucrose in the diluted juice .....	11.98
Per cent sucrose in the bagasse .....	2.90

- (1) Brix of the normal juice =  $19.21 \times 0.97$  ..... 18.63  
 Dilution per cent diluted juice  
       =  $100(18.63 - 14.52) \div 18.63 = (\text{See } 388)$  22.06  
 Dilute juice extraction =  $3450 \div 3254 \times 100 =$  ..... 106.02  
 Dilution per cent cane =  $106.02 \times 0.2206 =$  ..... 23.39  
 Mill extraction, normal juice per cent cane  
       =  $106.02 - 23.39 =$  82.63
- (2) Weight of bagasse =  $(3254 + 877) - 3450 =$  ..... 681 tons  
 Sucrose in the bagasse =  $681 \times 0.029 =$  ..... 19.745 tons  
 Sucrose in the extracted juice =  $3450 \times .1198 =$  ..... 413.31 tons  
 Sucrose in the cane =  $413.31 + 19.745 =$  ..... 433.055 tons  
 Mill extraction, sucrose extracted per cent sucrose in cane,  
        $413.31 \div 433.055 \times 100 =$  ..... 95.44

## Résumé:

Dilute juice extraction, per cent cane.....	106.02
Dilution, per cent cane.....	23.39
Extraction, normal juice per cent cane.....	82.63
Extraction, sucrose extracted per cent sucrose in the cane.	95.44

**379. Dry Milling Extraction Number.** (*For Mill Adjustments.*)—Considerable information of value concerning the performance of the various mills in a tandem may be obtained from the analysis of the bagasse as it passes from one mill to the next without saturation. A section of the bagasse blanket is marked in any convenient way (a boundary of dark oil, colored stain, lime, or the like) and samples are taken from this section as it emerges from the different mills. The bagasse from each mill is analyzed. The extraction of each mill may be calculated from the fiber as follows: Juice extracted per cent cane = (per cent fiber in bagasse — per cent fiber in cane)  $\div$  per cent fiber in bagasse, times 100.

**380. Saturation. Maceration. Imbibition.**—The quantity of water used in saturating the bagasse can only be determined with certainty by weighing or by accurate measurement. An estimation of the water is sometimes made by an inferential method similar to that used in estimating the weight of the cane. Inferential methods for the water are open to the same objections as those for the weight of the cane.

The water should be actually weighed and its percentage on the cane calculated or it should be measured and calculated over to weight. The percentage should be in terms of the weight of the cane.

In Hawaii the dilution is expressed in terms of the *normal* juice instead of the dilute juice as in Cuba and Porto Rico.

Dilution per cent Normal Juice = (Brix Normal Juice — Brix Dilute Juice)  $\div$  Brix dilute juice times 100.

Saturation per cent cane = weight of the water  $\div$  weight of the cane  $\times 100$ .

There was formerly much confusion among sugar-chemists in the method of stating the amount of saturation-water used. Dr. Spencer suggested the adoption of the expression "Per cent dilution" for the water as calculated from the density of the juices, since this represents the water, in terms of the total juice, that must actually be evaporated on account of the use of saturation. For the saturation-water as calculated from its weight and that of the cane, the expression "Per cent saturation" or "maceration" was suggested. Today these two expressions as above understood are almost universally used in sugar factory practice.

#### CONTROL OF THE SUGAR-BOILING

**381. Control of Vacuum-pans and Crystallizers.**—The control of the vacuum-pans and crystallizers requires rapid analytical work of moderate accuracy.

The analysis of the sirup as made in the daily routine work, or in its stead the analysis of the juice, and that of the molasses indicate the quantity of the latter to be drawn into the pan to produce a massecuite of the desired purity. If a cut strike is to be boiled, the purity of the above massecuite and that of the molasses to be boiled-in, supply the data for calculating the quantity of each of these required to produce a massecuite of a certain purity. These calculations are made by the following formula, with sufficient accuracy for the purpose:

Let 100 = total weight of massecuite in the strike;

$P$  = purity of the sirup, or, in the case of a cut strike, that of the massecuite left in the pan;

$p$  = purity molasses to be boiled-in;

$M$  = purity of the required massecuite;

$x$  = percentage by weight of that part of the strike to be formed of molasses;

$100 - x$  = percentage of the strike to be derived from sirup or from a previous boiling;

$$\therefore x = \frac{100(P - M)}{P - p}.$$

The proportions of the materials used in making a mixture of a certain purity may also be quickly calculated by the diagram method, page 386. It is not feasible in pan-work to base the calculations on actual weights. The approximate densities of the massecuite footing, for example, and the molasses to be boiled in should, however, be considered.

A sample of the mixed massecuite should be brought to the laboratory immediately the strike is dropped. A portion of this should be dissolved in water to form a solution of about 15° Brix, and its apparent purity should be determined. A second portion should be purged in a laboratory centrifugal and the purity of the molasses be determined as above.

The labor of calculating the apparent coefficient of purity may be avoided by the use of the expanded Horne's table, page 494.

The purity data of the massecuites and molasses samples should be promptly sent to the superintendent and the pan-boiler. All mixed strikes, especially those of the lowest purity, should be controlled in this way. The relation between the purity of the massecuite and the molasses purged from it in the laboratory, immediately after boiling, is a valuable guide in boiling low-purity mixed strikes. A convenient centrifugal is shown in Fig. 130, and a filtering device in Fig. 131. This filter is inexpensive and very efficient. It is a copper funnel, separable at the ground joint, A, to facilitate cleaning. The filtering surface is of centrifugal lining-sieve having about 625 round holes per square inch. The sieve must be supported by braces. The funnel is used in connection with a vacuum-filtering flask. Connection is made with the vacuum system of the factory.

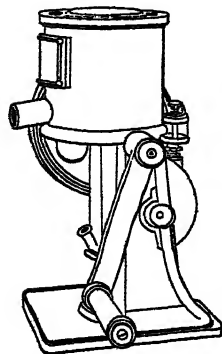


FIG. 130.—Laboratory Centrifugal.

Very often this method of control will indicate whether the pan-boiling is good or poor. When a low-purity massecuite yields a high-purity molasses, on immediate purging, it indicates poor boiling. A few days' experience with the pans, following the work with these control-tests, will usually indicate whether the sugar-boilers are obtaining the best results the pans and material are capable of yielding.

#### DIVISION OF THE SEASON INTO PERIODS

**382. Division of the Manufacturing Season into "Runs" or Periods.**—In order properly to present the laboratory and manufacturing data to the management of the sugar-house, the season should be divided into a series of short periods, or "runs." In a factory that is not operated on Sundays, the periods may conveniently end with that day, making a run of two weeks. In many factories it is customary to close a period's work when the machinery is necessarily stopped on account of bad weather or a mishap.

Dr. Spencer preferred to close a period arbitrarily, after a certain number of days' work, in order that the periods of one crop may be readily compared with those of previous years. In view of the very great labor involved in securing the data, making the calculations and preparing the reports, a period of two weeks is a convenient one. Moreover, this permits setting apart a certain day of the week for stock-taking.

If the machinery is idle, it is only necessary to measure the quantity of material in process at the various stations, and calculate their commercial sugar-value.

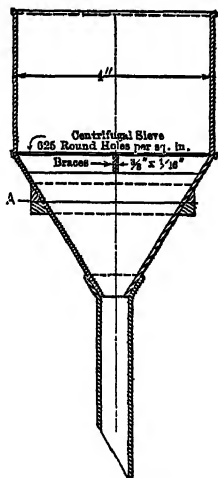


FIG. 131.—Massecuite Funnel for Separating the Molasses.

If the factory is in operation the following is the mode of procedure:

The chemist should prepare slips of paper with the numbers or other designations of the tanks and various pieces of apparatus, conveniently arranged for noting the measurements at the stations.

If the factory day ends at midnight, for example, the chemist, an assistant, and the laboratory helper, provided with sample cups, a measuring-rule and a thermometer should at that time go systematically through the factory drawing samples of the juice, etc., and measuring the quantity of material at the various stages of manufacture at the same time noting the temperature. This stock-taking should begin with the juice and end with the sugar, and must include all material in process, even the sugar in the centrifugals. It is usually more convenient to measure the depth of the empty space in the tanks, rather than that of the liquor. By using a very small measuring-cup for sampling, one cupful, for example, may be drawn from a quarter of a tank of sirup, two cupfuls from half of a tank and so on, thus forming a composite sample that will represent the average composition of the sirup with a fair degree of accuracy. If the tanks are of different sizes, the quantity drawn from each must be varied accordingly.

The volume of juice in the multiple effects should be estimated and may be considered a constant quantity from run to run, when the apparatus is in use. The material in the multiple effect may be measured when liquidated into tanks or the apparatus may be calibrated with water at the beginning of the season. The density of the sirup in the vessels of the evaporator should be ascertained from time to time as a guide in calculating the sugar value.

By prearrangement with the sugar boiler, certain tanks of sirup (*mela-dura*) and molasses may be omitted as such from the stock and be used to complete strikes of massecuite then in the pans. Thus these massecuites need not be measured until they reach the crystallizers or if immediately purged, the sugar and molasses may be separately considered. If this arrangement is not feasible, the sugar boilers should, at the whistle signal, note the depth of sirup and molasses in the tanks and indicate the approximate depth of massecuite in the pan by chalk marks. The condition of the massecuite should be noted or, preferably, proof-stick samples should be drawn for analysis. The quantity of sugars in the centrifugals, hoppers and bins should be noted, also the last serial package number.

Stock-taking in a large factory need require but a few minutes if the above scheme be followed. The results are practically as accurate as were the stock taken during a shut-down.

When the run report is called for to include a certain date, the work of stock-taking may be facilitated should it be known that the factory will be shut down a day in advance or a day later for cleaning or other reason. In this event the cane ground before or after the date is either carried as stock and figured to sugar or its product is deducted as the case may require.

383. Estimation of the Yield of Sugar for Stock in Process.—The samples having been taken as described, these are carried to the laboratory and analyzed for Brix, sucrose (polarization) and apparent purity. From suitable tables prepared for the various sized tanks in the factory the volume of each

of the different grades of material is calculated to convenient units (gallons, cubic feet or metric measurements as preferred). Formerly the available sugar in each grade of material in stock was calculated by yield formulae differing for the different grades. The methods now in use is similar to that employed for many years for calculating stock in refineries; namely to obtain the total of the solids, sucrose and impurities of whole stock and then calculate the available sugar and molasses from these data.

On a suitable "stock sheet" form the data as obtained should be entered, columns being provided for Grade of Material; Temperature, as observed in the factory ( $T$ ); Volume of Material (1); Brix of the material at the temperature  $T$  (obtained as described below); Weight per Unit Volume at this temperature (2); Weight of the Material  $[(1) \times (2) = (3)]$ ; Corrected Brix ( $17\frac{1}{2}^{\circ}$  or  $20^{\circ}$ ) (4); Weight of Solids  $[(3) \times (4) = (5)]$ ; Sucrose (polarization) (6); Weight of Sucrose  $[(3) \times (6) = (7)]$  Weight of Impurities  $[(5) - (7) = (8)]$ .

The Brix at the factory temperature  $T$  is not obtained by direct observation but by re-correcting the Corrected Brix (4) back to the factory temperature  $T$ . For example: Suppose the clarified juice in the factory to have a temperature of  $80^{\circ}\text{C}$  and a corrected Brix ( $17\frac{1}{2}^{\circ}\text{C}.$ ) of 15.20. By the table on p. 458,  $80^{\circ}\text{C}.$ , has a correction of 6.59. Then  $15.20 - 6.59 = 8.61$ , is the Brix at  $80^{\circ}\text{C}.$ , and the corresponding weight per unit volume may be found on p. 451. Similarly if the standard temperature is  $20^{\circ}\text{C}.$ , and the corrected Brix is at that temperature, the correction is obtained from the table on p. 469 and the weight per unit volume from the table on p. 459.

The totals for the weight of material (3), Solids (5), Sucrose (7), and Impurities (8), having been obtained, the average purity of the stock in process is obtained thus:

$$\frac{\text{Weight of Sucrose}}{\text{Weight of Solids}} = \text{average coefficient of purity of stock in process } (C).$$

Let  $x$  = yield of anhydrous commercial sugar in terms of the solids in the stock;

$C'$  = Purity of sugar produced in that period;

$M$  = Purity of Molasses produced in that period.

$$\text{Then } x = \frac{100(C - M)}{(C' - M)}. \quad (\text{See page 383.})$$

Weight of solids in Stock (5) multiplied by  $x$  equals weight of dry commercial sugar (9) which, divided by the per cent solids in the sugar for that period equals the weight of commercial sugar in stock (10).

Solids in Stock (5) minus Solids in Sugar (9) equals weight of solids in Molasses (11) which, divided by Brix of molasses equals weight of molasses in stock (12) multiplied by per cent sucrose in molasses equals weight of sucrose in molasses in stock. The weight of molasses (12) divided by the weight per gallon gives the gallons of molasses in the stock.

Having thus determined the weight of sugar in stock (10) and the weight of molasses in stock (12), these may be added to the sugar and molasses



actually made, thus giving "sugar made and estimated" and "molasses made and estimated" for the calculations of the various data, retention, yield, losses, etc., for the technical report. The usual practice is to calculate the figures "to date" and, by subtraction of previous "to date" figures, obtain the figures for the run.

#### LOSSES OF SUGAR IN MANUFACTURE

**384. Classification of Losses.**—The various losses may be classified into two general kinds (1) determined and (2) undetermined. The only losses which are determined and recorded in regular sugar-house control are those in the bagasse and filter-press cake and these losses are of the mechanical type, *i.e.*, the sugar is lost as such, in the form of sucrose.

**385. Determined Losses.**—*The Bagasse.*—The loss in the bagasse is calculated from the estimated weight of the material and its analysis.

If the weight of the saturation-water be known, the bagasse is the weight of the cane + weight of saturation-water - weight of the mixed dilute juice.

It may occur that the weight of the saturation-water is not known. In this event an inferential method must be used. Fiber per cent cane  $\times 100 \div$  fiber per cent bagasse = bagasse per cent cane. The uncertain quantity in this calculation is the percentage of fiber in the cane, due to the difficulty of sampling the cane. This latter method is generally employed in Hawaii.

*Filter-press Cake.*—The loss of sucrose is calculated from the analysis and weight of the press-cake.

The weight of the press-cake is usually estimated from the actual average weight of the contents of several filter-presses. When this is not practicable, the cake from a single chamber or frame of the press is weighed from time to time to obtain an average weight and this number is multiplied by the number of cakes in the press, to obtain the total weight. The cake weighs approximately 60-62 pounds per cubic foot.

There is a loss of sucrose in the juice absorbed by the filter cloth. This is usually included with the unknown losses. This quantity varies with the filter-press methods. It will vary from almost nothing with double filter-pressing to 0.5 pound or more per filter cloth.

**386. Undetermined Losses.**—(a) *Apparent Losses.*—These are not real losses, as the name implies, but they may play a large part in the reported losses of a factory. They are due to some error in cane, juice or sugar weights; or to errors of analysis or to incorrect stock estimates. These last will correct themselves in "to-date" figures. Apparent losses are reducible to a minimum by careful supervision of weights, tests and methods. It is equally evident that the same errors which cause apparent losses may, if operating the opposite way, cloak true losses and tend to make the factory figures appear much better than they really are.

(b) *Mechanical Losses.*—These include all waste of sugar in the form of sucrose. Besides the known losses in bagasse and press-cake enumerated above, there may be losses of sugar solutions to the sewer through leakage or spillage, though in a well-regulated factory such losses should be negligible.

Entrainment is a mechanical loss, the sugar being carried over from the vacuum apparatus to the condenser waters in the vapor in the form of minute bubbles ("Vesicular transference"). Modern apparatus practically eliminates this source of loss but a constant check should be kept on condenser waters (see p. 351).

The loss by entrainment may be estimated by Norris' table, page 442, from the analysis of the water flowing from the condenser and its weight as calculated from the temperature changes and the quantity of water evaporated. Detailed instructions are printed with the table.

(c) *Inversion and Decomposition Losses.*—The chemical nature of inversion has already been described (192.) It may be well to repeat that sucrose, when acted upon with acids or acid salts, changes into a mixture of dextrose and levulose called "invert sugar" (generally called "glucose" together with all other reducing sugars, and so called in this book). The extent of the inversion depends upon the nature of the acid (mineral acids having a much greater inverting power than organic acids); the quantity of the acid present in relation to the amount of sucrose: the temperature of the acid-sucrose mixture (increase of temperature speeds up the inversion greatly) and finally the time that the acid and sucrose are in contact. The hydrogen-ion concentration (pH) is a function of the first two factors (kind and amount of acid present) so the importance of pH control in the prevention of inversion losses is therefore evident.

The estimation of inversion in a sugar factory is a complicated matter because of the simultaneous decomposition of sucrose and glucose from the action of heat and alkalis. Glucose ratios (relationship between sucrose and reducing sugars 361) on the successive products are of value in indicating excessive inversion but the assumption in the use of such ratios is that no glucose has been decomposed.

*Prevention of Inversion Losses.*—Both inversion and decomposition losses can be largely prevented by a strict pH control on the clarification. Walten, McCalip and Hornberger<sup>4</sup> showed that the initial pH of the juice should be such that it will reach the sirup stage with a pH between 6.7 and 7.0. If the sulphitation process is employed, a lower pH may be safely carried. (See page 58.) Under these conditions inversion during clarification and subsequent evaporation and boiling can be reduced to a minimum and the decomposition of glucose due to excessive alkalinity will be avoided, so far as possible.

Glucose is usually destroyed to some extent in the manufacture, therefore it is advisable to figure a glucose balance as well as a sucrose balance to obtain light on the losses.

Changes in the saline coefficient are sometimes used in tracing losses either by inversion or mechanically. Zimmermann<sup>5</sup> suggests a method based upon the persistence of certain of the soluble salts throughout the manufacture. (See page 383.) He determines the sucrose by the Clerget method and the ash as sulphated ash. He transfers the ash to a beaker and precipitates the lime, etc., with ammonium oxalate in the presence of ammonia, then washes

<sup>4</sup> *Journal Ind. Eng. Chem.*, Vol. 17 (1925), 51.

<sup>5</sup> *Int. Sugar Journ.*, 1914, 16, 383.

out the soluble sulphates left in the precipitate and determines their quantity by difference. The calculations are illustrated by one of Zimmermann's examples:

Mill-juice: Sucrose, 10.5 per cent; soluble sulphates in the ash, 0.31 per cent;  $0.31 : 10.5 = 1 : x$  and  $x = 33.87$ , the ratio for juice. Sirup: Sucrose, 46.3 per cent; soluble sulphate in the ash, 1.38 per cent;  $1.38 : 46.3 = 1 : y$  and  $y = 33.55$ , the ratio for sirup. The change in the ratio from juice to sirup is  $33.87 - 33.55 = 0.32$ , corresponding to 0.32 per unit of sulphates. Then  $33.87 : 0.32 = 100 : x = 0.94$ , per cent loss on sucrose in the juice.

This is evidently a very exaggerated example. The soluble sulphates are much higher than the usual total ash and the loss is excessive. This method is quoted to call attention to this class of investigations.

### LABORATORY AND FACTORY RECORDS

**387. Test Books and Records.**—It is difficult to plan a set of books and forms without knowing something of the needs of the owners, the force of chemists available for control and whether this control is to be partial or fairly complete.

The usual reports include: (1) A slip for the Manager, Superintendent and Engineer, giving preliminary data of the mill work and control analyses of the juices, the output of sugar and the fuel consumption; this should be supplemented by frequent reports to the Engineer on the analysis of the bagasse. (2) The preliminary report should be followed by what may be termed an "operating report," which should include data covering the entire line of chemical control and the manufacture. This should include mill and manufacturing statistics. (3) Run reports at stated intervals, giving a résumé of all data collected both for the run and to date, including a sucrose balance and a statement of yield and losses. The data should be full enough to indicate the methods of manufacture and supply the owners a permanent record of methods. Working and lost time should also be reported to indicate what portion of the factory's capacity is being utilized. (4) Laboratory records: (a) Used in the analytical work. (b) Extraction figures, etc. (c) Records of pan-work. (d) Unit-book, used in recording the quantities of materials, products and by-products and in calculating weighted averages.

Printed forms should be supplied for the entries in the routine laboratory work. A large space should be provided in these forms for the figuring with a view to tracing errors. The use of printed forms also promotes systematic work. A loose-leaf binder is convenient for these forms and a sheet should be used each day. All figuring should be on the sheets or in special books and never on scraps of paper.

It is advisable to have printed forms for calculating and recording mill data, operating and lost time and fuel consumption. A special blank should be posted at the mills for reporting the delays and their causes. These figures should be tabulated from time to time for the use of the General Manager and the Chief Engineer.

The daily laboratory reports, for a fairly complete control, should include:

(1) Analyses of the diluted, normal (calculated) and residual juices. (2) Fiber and sucrose in the cane. (3) Analysis of the sirup. The Brix is for the control of the evaporation and the purity coefficient for that of the defecation. (4) Analyses of the massecuites and molasses to control the injection of molasses and the work of the crystallizers. (5) Moisture and polarization of the sugars and occasionally ash tests. The moisture has a bearing on the storage qualities of the sugar; the polarization must meet market requirements; the ash is an additional check upon the purification of the juice. (6) Analysis of the final molasses to meet market conditions and to control the pans, crystallizers and centrifugals. (7) Analysis of the filter-press cake to control the loss of sugar. (8) Analyses of the bagasse at frequent intervals, including moisture, fiber, and sucrose tests, for mill control. (9) Frequent examination of the feed-water for sugar, for the protection of the boilers. (10) Entrainment tests in the condenser water, to protect against carelessness in the evaporation and in the pan-boiling.

The manufacturing data that should be included in this report are records of the cane ground per hour and per day; operating and lost time; saturation and dilution; mill extraction; available sugar, sugar in process and sugar dried; fuel consumption. These figures should be for the day and to date.

A convenient arrangement of a run report is to place the manufacturing data on the lefthand side of the sheet and the analytical data and figures derived from them on the right.

## CHAPTER XXXV

### SUGAR-HOUSE CALCULATIONS <sup>1</sup>

**388. Introductory.**—All materials to be dealt with by the chemist are composed of sucrose and non-sucrose, the latter including water, dextrose, levulose, organic non-sugars, (marc, etc.) and inorganic matter (ash). Certain of these substances persist throughout the manufacture, others through but one or two stages of it. A knowledge of the proportions of these substances in the original material, products and by-products, is the basis for the construction of algebraic equations, with which yields, quantities, capacities, etc., may be calculated.

For the purposes of the usual calculations, in addition to the proportions in which the various constituents are present, certain relations between the constituents themselves are often required, such as purity coefficient, saline coefficient, etc. The problems are very simple when a constituent of the original material passes practically unchanged through the processes, *e.g.*, the fiber in dry milling.

The following formula illustrates the principles involved in many of the sugar-house calculations.<sup>1</sup> The water used in saturating the bagasse in milling cane dilutes the extracted juice; the percentage of this dilution is ascertained by means of an equation based upon the fact that the solids (Brix) of the extracted normal (undiluted) juice are present in the diluted juice:

Let 100 = the weight of diluted juice;  
    *b* = degree Brix of diluted juice;  
    *B* = degree Brix of normal juice;  
    *x* = weight of dilution-water in diluted juice;  
100 - *x* = the weight of normal juice,

then

$$100b = B(100 - x) \text{ and } x = 100 - 100b/B = 100(B - b)/B.$$

This is the usual dilution formula and is used because it is the diluted juice that is weighed or measured. The value of *x* multiplied by the percentage of dilute juice extracted from the cane gives the dilution in terms of the weight of the cane.

In a similar way, calculations may be based upon the fiber of the cane, the dry matter of the press-cake, the ash or a constituent of it, etc.

Since many of the numbers ascertained in sugar analysis are not absolute, most of the results of sugar-house calculations based upon them are approxi-

<sup>1</sup> The mark "/" is used to indicate division.

mations, but are usually sufficiently accurate for the purposes of the manufacturing control. The prime requisite is that the analyses be conducted with such a degree of exactness that all results will be strictly comparable. These considerations apply especially to massecuites and molasses in whose analysis absolute results cannot be expected.

The full work of deducing the formula is usually given in the following paragraphs, with a view to assisting the beginner in the construction of formulæ that are not given or that are necessitated by special conditions.

**389. Dry-milling Formula.**—The fiber or marc is the constant:

Let 100 = the weight of the cane;

$B$  = the weight of the bagasse from 100 cane;

$F$  = the percentage of the marc in the cane;

$F'$  = percentage of marc in the bagasse;

$x$  = percentage of juice extracted;

$$(1) \ x = 100 - B; \ (2) \ F'B = 100F; \ \text{whence} \ B = 100F/F';$$

substituting the value of  $B$  in (1)

$$x = 100 - 100F/F' = 100(F' - F)/F'.$$

The similarity of this formula and that for calculating dilution (388) is noticeable. This should be expected since in the one the constant (Brix) is diluted and in the other the constant (marc) is concentrated, *i.e.*, its percentage relation to the bagasse as compared with cane increases.

**390. Dilution Formulæ.**—The formula for the dilution of the mill-juice in terms of the diluted or mixed juices has been given in the Introductory (388). To reduce this number and the following dilution number to percentage terms of the weight of the cane, it is necessary to multiply the values of  $x$  by the percentages of diluted and normal juice, respectively. The dilution per cent normal or undiluted juice is calculated as follows:

Let 100 = the weight of normal juice;

$B$  = the Brix of the normal juice;

$b$  = the Brix of the diluted juice;

$x$  = the percentage of dilution in terms of the normal juice,

then  $100 + x$  = the weight of the diluted juice, and since all the solids (Brix) of the normal juice are found in the diluted juice,  $b(100 + x) = 100B$  and  $x = 100B/b - 100 = 100(B - b)/b$ . This number is used in certain countries to indicate the quantity of maceration-water that has been used, though in fact it only indicates the part of the water that has passed into the juice.

**391. Concentration and Evaporation Formulæ.**—These formulæ are similar to those for dilution and are derived in the same way:

Let 100 = the weight of the juice, etc.;

$b$  = the Brix of the juice;

$B$  = the Brix of the concentrate;

$x$  = the percentage, by weight, of water evaporated;

then

$$100b = (100 - x)B, \text{ whence}$$

$$x = 100(B - b)/B. \text{ (See Table 25, page 439.)}$$

The percentage of evaporation by volume is derived as follows:

Let 100 = the volume of the juice, etc.;

$b$  = the Brix of the juice of  $g$  specific gravity;

$B$  = the Brix of the concentrate of  $G$  specific gravity;

$x$  = the percentage, by volume, of water evaporated;

then

$$GB(100 - x) = 100gb,$$

and

$$x = 100(1 - gb/GB),$$

the volume of water evaporated. (See Table 26, page 441.) Having two solutions of known degrees Brix,  $B$  and  $B'$ , to determine the degree Brix of a mixture composed of the volumes,  $V$  and  $V'$ , of these solutions having specific gravities of  $G$  and  $G'$ . Then,

$$x = \text{degree Brix required} = \frac{VBG + V'B'G'}{VG + V'G'}.$$

In cases where there is no great divergence in the specific gravities the formula may be used for practical purposes as follows:

$$x = \frac{VB + V'B'}{V + V'}.$$

Formula for the calculation of the water required, per cent by weight, to reduce a sugar solution of a given density to any required density.

$x$  = per cent of water required;  $B$  = initial degree Brix;  $b$  = brix after dilution;  $\frac{B - b}{B} = E$ , and  $\frac{100E}{1 - E} = x$ , the per cent required, which, by substitution for  $E$  becomes  $x = 100 \frac{(B - b)}{b}$ .

To determine the volume  $V$  of a sugar solution before concentration.

$b$  = degree Brix, and  $s$  = the specific gravity of the solution before concentration;  $B$  = degree Brix, and  $S$  = specific gravity after concentration to a volume of 100.

$$V = \frac{100SB}{sb}.$$

**391. Commercial Sugar Formulæ.**—(A) This formula has a wide application in the sugar-house control and in the estimation of the capacities required in crystallizers, etc. It employs the polarization of the primary material and gives the yield in terms of this material.

Let  $x$  = the percentage yield of commercial sugar of  $p$  polarization and  $S$  per cent dry matter;

100 = the weight of the primary material (massecuite, molasses, etc.)  
of  $P$  polarization and  $B$  per cent dry matter (Brix);

$M$  = coefficient of purity of the residual molasses;

then

$P - px/100$  = the weight of sucrose in the molasses;

$B - Sx/100$  = the weight of dry matter (Brix) in the molasses.

Since the coefficient of purity of a sugar material is the percentage of sucrose in its dry matter we have

$$M = 100 \frac{P - px/100}{B - Sx/100};$$

clearing of fractions, transposing and reducing,

$$x = \frac{100P - BM}{p - SM/100}, \quad (A)$$

the yield of commercial sugar. If the product is refined sugar of 100° polarization, as is customarily assumed in refinery work, the formula reduces to

$$x = \frac{100P - BM}{100 - M}.$$

(B) This formula uses the purity instead of the polarization as above and is applicable in the calculation of the yield of sugar from massecuites, molasses, etc. It also gives percentages in terms of the primary material.

Let  $x$  = the percentage of commercial sugar of  $C'$  purity coefficient and  $S$  per cent of dry matter;

100 = the weight of primary material,  $C$  its coefficient of purity and  $B$  its degree Brix;

$M$  = the coefficient of purity of the residual molasses.

Then  $BC/100$  = weight of sucrose in the material;

$Sx/100$  = the weight of dry matter in the sugar;

$$\frac{Sx}{100} \times \frac{C'}{100} = \frac{C'Sx}{10,000}, \text{ the weight of sucrose in the sugar;}$$

$B - Sx/100$  = the weight of solids in the molasses;

$$\frac{BC}{100} - \frac{C'Sx}{10,000} = \text{the weight of sucrose in the molasses.}$$

As in the preceding sugar formula, an equation based upon the coefficient of purity of the residual molasses is formed:

$$M = 100 \left( \frac{BC}{100} - \frac{C'Sx}{10,000} \right) \div \left( B - \frac{Sx}{100} \right),$$



clearing of fractions, transposing and reducing, we have

$$x = 100 \frac{C - M}{C' - M} \times \frac{B}{S}^2 \quad (B)$$

(C) This formula has the same applications as the preceding. It is derived the same way, but is based upon moisture-free materials:

Let  $x$  = the percentage yield of anhydrous sugar in terms of the dry matter (Brix) in the primary material, and let the other letters have the same meaning as in the previous formula (B).

Then  $100 - x$  = the dry matter (Brix) of the residual molasses;

$C'x + M(100 - x) = 100C$ , whence  $x = 100 \frac{C - M}{C' - M}$ , the percentage of anhydrous sugar in terms of the dry matter in the primary material. This formula is used for calculating available sugar in stock in process in raw sugar work. (See page 374.) When the product has a polarization of 100 (sucrose) the formula then becomes  $x = 100 \frac{C - M}{100 - M}$  which is the formula used in refineries to calculate the yield of refined sugar from the purity of a massecuite and its resulting sirup (molasses).

<sup>2</sup> The previous formula (A) will be seen to be identical with the one above except that the first employs the polarization and the Brix, and the second the purity and Brix, of the primary material and of the commercial sugar. The polarization—Brix—purity relationship

$$\frac{100P}{B} = C \quad \text{and} \quad \frac{100p}{S} = C'$$

gives  $P = \frac{BC}{100} \quad \text{and} \quad p = \frac{SC'}{100}.$

Substituting these values in the formula

$$x = \frac{100P - BM}{p - SM/100},$$

we have

$$x = \frac{\frac{100BC}{100} - BM}{\frac{SC'}{100} - \frac{SM}{100}}.$$

Clearing of fractions, we have

$$x = 100 \frac{(C - M)}{(C' - M)} \times \frac{B}{S}$$

which is the second formula (B).

(D) Another widely used available sugar formula deals with purities only and gives the yield of sucrose in terms of the sucrose present in the primary material.

Let  $x$  = the yield of sucrose present in the commercial product, per cent of the anhydrous sucrose ( $C$ ) present in the original material and let all other letters have the same significance as before.

It is evident that this resolves itself into the previous formula multiplied by the sucrose per cent dry matter (*i.e.*, the purity) of the sugar ( $C'$ ) divided by the sucrose per cent dry matter (purity) of the primary material ( $C$ ) or

$$x = 100 \frac{C'(C - M)}{C(C' - M)}.$$

When  $C'$  becomes 100 (*i.e.*, the product is sucrose) the formula then is

$$x = \frac{100(C - M)}{C(100 - M)} \times 100.$$

This formula is sometimes called the "drop in purities" formula.

The above formulæ may be applied in the calculation of the yield of sugar from the cane or juice. In fact it has been shown on page 360 that the last formula given corresponds to the theoretical retention figure of the Winter-Carp-Geerligs formula  $[100(1.6 - 40/C)]$  if the purity 23.57 is substituted for  $M$ . The "available sugar" formulæ are more widely used for such estimates. These formulæ find their chief uses in calculating the yield of sugar in process in massecuites, etc.

**392. Crystallizer Capacity.**—The commercial sugar formulæ may be used in estimating the capacity required in certain machinery, notably pans, crystallizers and molasses-tanks, using true solids and sucrose in accurate work.

Let it be required to estimate the quantity of crystallizer massecuite of 94° Brix and 60° purity that would be produced from clarified juice of 20° Brix, 18 per cent sucrose and 90° purity, the sugar that has been extracted having an analysis of 96° polarization and 99 per cent dry matter: Using formula (A), 391, we have

$$x = \frac{(100 \times 18) - (20 \times 60)}{96 - (99 \times 60/100)} = 16.39$$

sugar per cent of the weight of the juice. Let the juice be 80 per cent of the weight of the cane, then  $16.39 \times .80 = 13.11$  sugar per cent cane. The sugar contained 99 per cent dry matter, therefore  $13.11 \times .99 = 12.98$  per cent dry sugar. The juice contains  $20 \times .80 = 16$  per cent dry matter (Brix solids)

\* This formula was developed by Noël Deerr who used the letters  $s$ ,  $j$ , and  $m$ , for the symbols, giving  $x = 100 \frac{s(j - m)}{j(s - m)}$ , and it is frequently known as the  $s,j,m$ . formula of Deerr. ("Cane Sugar," Manchester, 1911, p. 509.)

on cane and  $16 - 12.98 = 3.02$ , the percentage of dry matter on cane going into the crystallizer massecuite;  $3.02 \div .94 = 3.21$ , the massecuite per cent cane. Massecuite of  $94^\circ$  Brix weighs 94.4 pounds per cubic foot, therefore  $3.21 \div 94.4 = 0.034$  cubic feet massecuite per 100 pounds cane, or 0.68 cubic foot per ton of cane. Massecuite swells considerably, owing to the crystallization of its sugar and the decomposition of certain salts (*see* page 101) and it may also be diluted from time to time (*see* page 98). An increase of volume of 25 per cent is safe allowance for alteration, or  $0.68 + 25$  per cent = 0.85 cubic feet. Further, the massecuite should remain in the crystallizer about four days, the time depending upon the size and type of the crystallizer, so we then have  $0.85 \times 4 = 3.4$  cubic feet per ton of cane daily milling capacity. If the milling capacity is 1850 tons of cane the crystallizer capacity should be  $3.4 \times 1850 = 6290$  cubic feet. The size of the crystallizers will depend upon the size of the vacuum-pans and under the usual conditions with pans of 12 feet in diameter striking 1000 cubic feet, would be approximately  $1000 + 25$  per cent = 1250 cubic feet;  $6290 \div 1250 = 5+$ , the required number of crystallizers to actually hold the massecuite. Allowance must also be made for one empty crystallizer to receive massecuite and one to be discharging to the centrifugals, or in all 7 crystallizers of about 1250 cubic feet gross capacity each.

This estimate is based upon juice of exceptionally high purity. In actual practice the estimates should be upon the juice of the lowest purity that is liable to prevail over an extended period at any time of the manufacturing season. The lower the initial purity, the larger will be the quantity of crystallizer massecuite. In actual estimates the true solids and purity should be used to avoid errors.

**393. Mixed Massecuites. Proportion of Molasses and Sirup.**—These massecuites should be boiled to a definite purity, depending upon that desired in the molasses to be obtained from them. This formula assumes that the densities of sirup and molasses are the same. This is sufficiently accurate for practical purposes:

(A) Let 100 = total weight of massecuite in the strike;

$P$  = purity of the sirup;

$p$  = purity of the molasses to be boiled-in;

$M$  = purity of the required massecuite;

$x$  = percentage by weight of the strike to be formed of molasses;

$100 - x$  = percentage by weight of the strike to be derived from sirup,

then

$$x = \frac{100(P - M)}{P - p}.$$

This formula may be applied with less accuracy when  $P$  is the purity of a footing or nucleus upon which a strike is to be completed with molasses.

(B) This calculation may be made with greater facility by Cobenze's<sup>4</sup> method for mixtures, illustrated in the diagram and example, Fig. 132. Let

<sup>4</sup> A. Cobenze, *Compendium der prakt. Photographie*, 9th ed., p. 379; Van Nostrand's Chemical Annual, 1913, p. 563.

85 be the coefficient of purity of a sirup and 55 that of a molasses and let it be required to make a massecuite of these having a purity of 65. Arrange the numbers as in the diagram. Subtract the purity number for the massecuite from that of the sirup and the number for the molasses from that of the massecuite and arrange the remainders as shown in the diagram; the remainder, 10, is then number of parts of sirup required and 20 that of the molasses. If the percentage of each constituent is required, divide the number of parts of each by the total number of parts, and multiply by 100. As in the previous method, using purities only in calculating the mixture, it must be assumed that the densities of the solutions are the same.

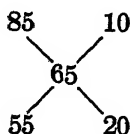


FIG. 132. — Cobenze's Diagram for Calculating Mixtures.

This method may be used for all mixtures and facilitates the solution of many otherwise complicated problems. If any three numbers used in the diagram are given the other two are readily ascertained.

394. Calculation of Added Water in Double Filter-pressing of Scums (Cachaza).—This is a problem in dilution and may readily be solved by the formula in 390 for dilution in terms of the normal juice. Let  $B$  of the formula represent the solids in the first press-cake,  $b$ , the solids in the reduced cake, and  $x$ , the percentage of added water, then  $x = 100(B - b)/b$ .

The following example will illustrate the calculation:

A press-cake contained 35.06 per cent dry matter and after reduction with water to a cream this contained 14.24 per cent dry matter:

$$x = 100(B - b)/b = 100(35.06 - 14.24)/14.24 = 146.2 \text{ per cent.}$$

395. Volume of Juice at a Given Temperature to Weight at Standard Temperature ( $17\frac{1}{2}^{\circ}\text{C.}$  or  $20^{\circ}\text{C.}$ ).—A sugar solution does not expand on heating at the same rate as water (*see* Gerlach's table, page 439). The expansion also varies with the sugar content of the solution. For the usual purposes the table of Gerlach may be used, but the following method gives practically the same results and is more convenient. Note the temperature of measurement; if the standard temperature is  $17\frac{1}{2}^{\circ}\text{C.}$  reduce the degree Brix of the juice to that at the temperature of measurement by means of Gerlach's table of corrections, page 458. Apply the specific gravity number corresponding to this reduced Brix to the weight of the cubic foot or gallon of water at the temperature of measurement.

Example: Required the weight of a cubic foot of juice of 15° Brix at  $17.5^{\circ}\text{C.}$  /  $17.5^{\circ}\text{C.}$  measured at  $28^{\circ}\text{C.}$

Degree Brix of the juice at $17.5^{\circ}\text{C.}$ .....	15.0°
Hydrometer correction (page 458) for 15° Brix at $28^{\circ}\text{C.}$ .....	.7

Degree Brix and corresponding specific gravity at $28^{\circ}\text{C.}$ .....	
(Sp. Gr. = 1.05831) .....	14.3°

Referring to the table on page 428, a cubic foot of water measured at  $28^{\circ}\text{C.}$  weighs 62.1289 pounds, therefore  $62.1289 \times 1.05831 = 65.75$  pounds, the

weight of juice required. Similarly, to correct to 20° C. the tables on p. 469 may be used. The error of this simple method is well within the limits of accuracy of tank measurements. It is preferable that all measurements be made at, as nearly as is practicable, the temperature of graduation of the hydrometers, thus keeping all errors at a minimum.

**396. Calculations Based upon the Relation between the Ash and Sucrose, etc.**—These methods are used in ascertaining whether sugar or other matters are destroyed or decomposed and removed in a boiling process, for example. The mineral constituents of the materials are assumed to remain unchanged during the process, therefore a comparison of the saline coefficients (362) should show whether sucrose has been destroyed, and similarly whether other constituents have been decomposed. A method of this class must be used with great caution, since very slight inaccuracy of analysis or loss of mineral matter may lead to an erroneous conclusion. The true or Clerget number should be used in sucrose comparisons.

Example (all sucrose numbers are by the Clerget method): A clarified juice containing 15 per cent sucrose, 0.3 per cent ash and 50 saline coefficient was evaporated to sirup containing 50.7 per cent sucrose, 1.02 per cent ash and a saline coefficient of 49.71. The reduction of the saline coefficient by  $50 - 49.71 = 0.29$ , indicates decomposition of sucrose. This loss in percentage terms of the sucrose in the juice is calculated by finding what per cent 0.29, the decrease in the saline coefficient, is of the original coefficient 50, in this case 0.58.

This method has long been used in beet- and cane-sugar manufacture in estimating losses, but its application is somewhat limited by the fact of the deposition of a part of the ash constituents on the evaporating surface and in storage tanks. C. H. A. Zimmermann<sup>5</sup> proposes to utilize only the soluble sulphates of the sulphated ash in this method, since the salts from which they are formed persist through the manufacture. The material is ashed as usual with the addition of sulphuric acid. The ash is transferred to an ashless filter or an alundum filtering-crucible and the soluble matter is removed by washing with hot water. The residue is dried and weighed. The difference between the original weight of ash and this residue is the weight of the soluble sulphates. The calculations are made as before.

<sup>5</sup> Int. Sugar J., 16 (1014), 338, also this work, Sec. 386.

## CHAPTER XXXVI

### PURCHASE OF CANE ON A BASIS OF ITS ANALYSIS

**397. General Considerations.**—Cane in Cuba is usually sold to the factories at a "flat" price, *i.e.*, its quality, as regards sucrose content and the purity of the juice, is not considered. A system of purchase on test would undoubtedly increase the profits of both the manufacturer and the grower. The grower would be forced to take better care of his fields and deliver the cane as soon as possible after cutting. Prompt delivery of the cane would result in increased delivery weight and sugar production with a decrease in costs to the manufacturer.

The problem of devising a system of purchase that will be not only equitable, but that may be operated without exciting the distrust of the farmer is not a simple one.

Sales are sometimes based upon the degree Brix of the juice, without regard to either the sucrose content or the coefficient of purity. This method forces the factory to pay a very conservative price for the cane, to offset the low purity of the juice in the early part of the season. This method has probably been used on account of the fairly good idea of density (Baumé) held by cane farmers in general.

The real difficulty in devising a system of purchase lies in the sampling of the cane itself. Small samples from a cart or car are usually of very little value in indicating the analysis of the cane and especially so when the juice for testing is expressed by a laboratory mill. The only method of sampling that has given practical results is that of the juice drawn from the factory mills and representing the entire load of cane. This method is included in the daily routine of many factories in comparing canes from the various fields and railway switches.

Given a representative sample of the juice from a particular lot of cane, the next step is the estimation of its sugar value to the manufacturer. This might be based upon the sucrose content of the cane, but the difficulty of determining this number is an objection. The method selected must be better than a rough approximation in order to protect the manufacturer and be fair to the farmer. Obviously, to be equitable, the method must be based upon a consideration of the available sugar, and in figuring this number, the coefficient of purity of the juice and the efficiency of the factory must enter. The method described in the following paragraph, though apparently complicated, is in fact quite simple.

**398. Available Sugar as a Basis of Cane Purchase.**—A special chemist and assistants are required for these tests.

*Reduction Factors.*—The chemist should first determine two reduction factors, in cooperation with the chemist of the factory, as follows: (1) Factor for reducing the degree Brix of the juice from the crusher to that of the mixed normal juice of all the mills. (2) Factor similar to the preceding for reducing the per cent sucrose of the crusher-juice to that of the normal juice. The first factor has long been used and is regularly determined at intervals by the factory chemist (341). To determine these factors, operate the milling plant for a short period without saturation-water; sample and separately analyze the juice from the crusher and the mixed juice from the entire plant. Calculate the ratio or factors, (1) Brix of the normal mixed juice  $\div$  Brix of the crusher-juice and (2), Sucrose per cent in normal juice  $\div$  sucrose per cent crusher-juice.

*Sampling.*—The number of the cart or car of cane is noted and the chemist is given a ticket bearing this number and is advised to prepare for sampling. If cart cane is to be sampled, it is advisable to group a number of loads under one test. The chemist notes the position of the cane on the conductor or elevator, using markers if need be, and after the cane reaches the crusher he allows a definite interval for the expressed juice to wash the rolls and then preferably samples continuously and automatically or at brief intervals during the passage of the cane. The sample is sent to the laboratory with the ticket corresponding to it. Sample follows sample in this way.

*Analysis.*—On receipt of the samples by the laboratory, the samples and tickets are arranged in regular order. A numbered cylinder is filled to overflowing with the strained juice and the remainder of the sample is held in reserve pending the completion of the test. The cylinders should be arranged in convenient groups, of ten, for example, and after the latest to arrive has been standing ten minutes the hydrometers are inserted. After a further interval of five minutes the degree Brix and temperatures are to be noted. The observed Brix, temperature and corrected Brix should be entered on a duplicate of the load ticket. These operations should be conducted very systematically, allowing a definite period for each.

The sucrose test should be made by Horne's dry lead method, page 314. The laboratory equipment should include a considerable number of 200 cc. (approximate) cylinders like *C* of Fig. 75. Each of these should have a mark etched on it to indicate approximately the 100 cc. point. A spoon having a conical bowl should be provided for measuring the dry lead. It should hold about 1 gram of lead, "struck" measurement.

Arrange a group of these cylinders, corresponding to those used for the density determination, and fill each to the mark with juice. Add a measure of lead to the juice, cover the cylinder with the palm of the hand and mix its contents by vigorous shaking. Filter, polarize and calculate the sucrose by Schmitz's table, page 481. Enter this test on the ticket.

*Available Sugar.*—Multiply the degree Brix by the corresponding reduction factor and the per cent sucrose by its factor. This gives the degree Brix and the per cent sucrose in terms of the normal juice. Calculate the coefficient of purity. The available sugar is now calculated by the method given on page 360, and this in turn to a basis of the factory by applying the efficiency and

normal juice extraction-numbers. The following example indicates the steps in the calculation:

Example:

Factor for reducing the degree Brix.....	0.978
Factor for reducing the per cent sucrose.....	0.965
Degree Brix of the crusher-juice.....	18.0
Per cent sucrose in crusher-juice.....	15.7
Assumed mill extraction, normal juice .....	78.0
Average efficiency number of the factory.....	98.0
Degree Brix of calculated normal juice..	$= 18.0 \times 0.978 = 17.60$
Per cent sucrose calculated normal juice.	$= 15.7 \times 0.965 = 15.15$
Coefficient of purity calculated normal juice.....	$= 15.15 \div 0.1760 = 86.08$
Sucrose extracted by the mills in normal juice.....	$= 15.15 \times 0.78 = 11.82\%$ cane

Referring to the second section of the table on page 488 we find 97.43, the yield number of 96° sugar corresponding to 86.1 purity. This number multiplied into the sucrose extracted gives the yield of sugar per cent cane on a basis of 100 factory efficiency,  $11.82 \times 97.43 = 11.52$ . Since the factory efficiency number is 98 we must reduce the yield figure accordingly:  $11.52 \times 0.98 = 11.29$ , the percentage yield of 96° sugar that may be expected from the cane.

The reliability of this method may be judged from the actual sugar-house results given on page 361.

This method enables the manufacturer to arrange a scale of prices based upon the actual yield of sugar that the cane may be expected to give in his factory.



## CHAPTER XXXVII

### ANALYSIS OF LIMESTONE, LIME, SULPHUR AND SULPHUROUS ACID

#### ANALYSIS OF LIMESTONE

**399. Preparation of the Sample.**—Fragments should be chipped from a large number of pieces of the stone and reduced to a uniform size, then mixed and sub-sampled by quartering. The small sample should be reduced to a very fine powder in an iron mortar or on a grinding-plate. Particles of metallic iron, from the mortar or plate, should be removed by stirring the powder with a magnet. Sift the powder through an 80-mesh sieve, and mix it thoroughly by sifting or otherwise.

**400. Determination of Moisture.**—Dry 2 grams of the powdered stone to constant weight in a tared flat dish or a watch-glass. The oven should be heated to  $110^{\circ}\text{C}$ . The loss of weight divided by 2 and multiplied by 100 is the percentage of moisture.

**401. Determination of Sand, Clay, and Organic Matter.**—Treat 1 gram of the powdered limestone in a beaker, with a few cubic centimeters of hydrochloric acid, being cautious, in adding the acid, to prevent the projection of particles of the material from the glass. Cover the beaker with a watch-glass and heat the liquid a few minutes. Collect the residue on a tared quantitative filter, wash it thoroughly with hot water, and reserve the filtrate (A) for further treatment. Dry the filter and residue to constant weight at  $110^{\circ}\text{C}$ . The weight of the residue multiplied by 100 is the percentage of sand, clay, and organic matter. Place the filter and residue in a tared platinum crucible and incinerate. The weight of this residue (A) multiplied by 100 is the percentage of sand and clay, *i.e.*, silica and combined silica and alumina. The difference between this percentage and that obtained before incineration is the percentage of organic matter.

**402. Determination of Soluble Silica.**—Evaporate the filtrate (A) from the preceding determination to strict dryness, on the water-bath, using a platinum or porcelain dish. Moisten the residue with hydrochloric acid and again evaporate to dryness. It is advisable to continue the heat for an hour or longer after apparent dryness, to insure the insolubility of the silica. Treat the residue with dilute hydrochloric acid; collect the insoluble portion on a small quantitative filter and wash it thoroughly with hot water until free of chlorides. Reserve the filtrate (B) for further use. Partially dry the filter and contents, then insert them in a tared platinum crucible, and char the paper by the application of a very gentle heat. If charred too rapidly, there may be

difficulty in subsequently burning off the carbon. Increase the heat until the filter is completely incinerated, and then raise it to bright redness. Cool the crucible and contents in a desiccator and weigh. The weight of the ash of good quantitative filters, or of the so-called "ashless filters," is so small that it need not be taken into account.

The weight of the residue, multiplied by 100, is the percentage of silica,  $\text{SiO}_2$ , in the soluble silicates of the stone.

**403. Determination of Total Silica.**—Mix the residue *A* (401), in the platinum crucible with four or five times its weight of <sup>1</sup> mixed carbonates of sodium and potassium and fuse at a red heat. Continue the heat about thirty minutes after the contents of the crucible are in a quiet state of fusion.

Remove the bulk of the mass from the crucible, while still warm, with a platinum wire, to facilitate the subsequent solution. Place the crucible and the material removed from it in a beaker and treat with dilute hydrochloric acid, being careful to avoid loss by the projection of the liquid from the glass. Use heat, if required. Wash and remove the crucible. Filter the solution and evaporate it to strict dryness, as described for soluble silica in the preceding paragraph. Treat the residue with dilute hydrochloric acid as before, wash it on a filter and reserve the filtrate (*C*). Incinerate the filter and heat the residue to bright redness, weigh, and calculate the percentage of silica as described in the preceding paragraph. Subtract the percentage of soluble from that of the total silica, to obtain the percentage of silica present in insoluble silicates.

**404. Determination of Iron and Alumina.**—Combine filtrates *A*, *B*, and *C* from the preceding operations and concentrate them to a convenient volume. Add a slight excess of pure ammonia to the solution while it is still hot, boil it until only a slight odor of ammonia can be detected, collect the precipitate on a small filter, filtering rapidly while the solution is hot. If there is considerable iron and alumina present, it is advisable to dissolve the precipitate with dilute hydrochloric acid and reprecipitate it with ammonia as directed above, uniting the filtrates (*D*). Partly dry both filters, and incinerate as advised for silica.

The residue consists of the mixed oxides of iron and alumina ( $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ). Multiply the weight of the residue by 100 to obtain the percentage.

It is not usually necessary to determine the iron and alumina separately. If required, however, proceed as follows: Treat 1 gram of the powdered limestone with concentrated hydrochloric acid, most conveniently in a platinum dish. Evaporate to strict dryness, moisten with hydrochloric acid, and again dry on the water-bath, as described for the silica determination. Treat the residue with dilute hydrochloric acid, with heat, and filter; wash the filter with hot water and treat the filtrate with ammonia, as described above, to precipitate the iron and alumina. Wash the precipitate into a small dish, dissolve it in sulphuric acid, and evaporate the solution nearly to dryness. Wash the residue into an Erlenmeyer flask, being cautious in the use of the water.

<sup>1</sup> Use strictly chemically pure, dry carbonates of sodium and potassium, mixed in molecular proportions and finely powdered. The proportions are 106 parts sodium carbonate to 138 parts potassium carbonate.

The iron is now most conveniently determined by titration with a standardized solution of potassium permanganate.

Add a small quantity of pure zinc-dust to the solution in the flask, to reduce the iron from the ferric to the ferrous state, and titrate with the decinormal permanganate solution. This solution is added until a faint permanent pink color is produced. Multiply the burette reading by .008 to ascertain the weight of ferric oxide in 1 gram of the stone, and this weight by 100 to obtain the percentage of ferric oxide ( $\text{Fe}_2\text{O}_3$ ); subtract this per cent from the combined percentages of iron and alumina, to obtain the percentage of alumina.

**405. Determination of Calcium.**—To the filtrate from the iron and alumina determination (*D*), corresponding to 1 gram of the stone, add sufficient hydrochloric acid to render it slightly acid. Concentrate this solution to about 250 ml., neutralize it with ammonia, heat to boiling, and add an excess of boiling-hot ammonium oxalate solution. Digest on the water bath for about one hour (or set aside for twelve hours) until the precipitate has completely settled out leaving a clear supernatant liquid. Decant through a filter paper, washing the precipitate two or three times by decantation with hot water containing a little ammonium oxalate and hydroxide. Then collect the precipitate of calcium oxalate on a quantitative filter, wash with cold water (filtrate *E*), dry and incinerate the filter in a tared platinum crucible, then ignite the residue strongly. The residue consists of almost pure calcium oxide ( $\text{CaO}$ ), and may be weighed as such.

If the limestone contains a considerable amount of magnesium the precipitate should be redissolved on the filter by warm dilute hydrochloric acid and the calcium reprecipitated by neutralization with ammonia and the addition of 1 or 2 cc. of ammonium oxalate solution. Filter, wash with cold water (add filtrate and washings to *E*) dry and incinerate the filter as before and ignite the residue strongly. The weight of  $\text{CaO}$  multiplied by 100 gives the per cent of calcium oxide in the stone. This value multiplied by 1.7847 gives the per cent of calcium carbonate.

The ignited calcium oxide in this determination combines with water and carbon dioxide very readily, hence cooling and weighing should be done as rapidly as possible.

**406. Determination of Magnesium.**—To the filtrate *E*, from the calcium determination, after concentration to approximately 100 ml., add a slight excess of ammonium hydroxide, chill the solution, then add sodium phosphate solution in excess, drop by drop, with vigorous stirring. After fifteen minutes add a decided excess of ammonia. Set aside during several hours, preferably overnight, to insure a complete precipitation. Collect the precipitate in a Gooch crucible, wash it with dilute ammonium hydroxide, containing 1 part  $\text{NH}_4\text{OH}$  of 0.96 specific gravity, to 3 parts water. The washing should be continued until free from chlorides. The precipitate is ammonium-magnesium phosphate; dry it, first at a gentle heat, then increase the temperature to expel the ammonia, and finally ignite it a few minutes in the flame of a blast-lamp to convert the residue into magnesium pyrophosphate. Cool the residue in a desiccator and weigh it. The weight of the magnesium pyrophosphate ( $\text{Mg}_2\text{P}_2\text{O}_7$ ) multiplied by .36208 gives the corresponding weight of magnesium

oxide. The magnesium is present in limestone as carbonate. Multiply the weight of the pyrophosphate by .7573 and the product by 100 to ascertain the percentage of magnesium carbonate in the stone.

**407. Determination of Carbonic Acid.**—It is not usually necessary to determine the carbonic acid, as it may be calculated from the quantity required to combine with the lime and magnesia, except when sulphates are present.

The gravimetric determination is made with one of the various forms of alkalimeters. Knorr's apparatus, Fig. 133, is one of the best of these. The method of using this apparatus is as follows: A weighed quantity, 5 grams or more, of the finely powdered limestone, is introduced into the flask *A* with 50 ml. or more of distilled water. The tube *G* is connected with a filter-pump to draw a current of air through the apparatus during the entire process. The

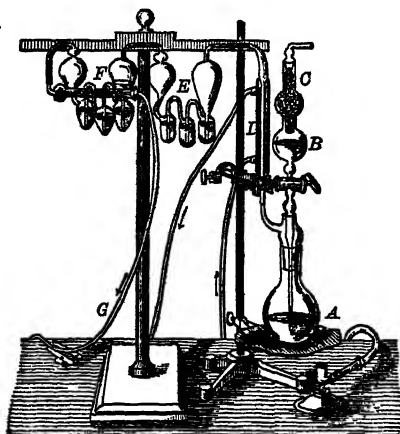


FIG. 133.—Knorr's CO<sub>2</sub> Train.

bulb *B* contains the acid for decomposing the stone, preferably concentrated hydrochloric. The guard-tube *C* is filled with fragments of caustic soda, potash, or with soda-lime, to prevent the entrance of carbonic acid with the air. Open the stop-cock on the bulb-tube *B* and admit the acid slowly; the liberated gas passes through the condenser *D*, where most of the moisture is condensed, thence through the bulbs *E*, containing concentrated sulphuric acid, which removes every trace of water; the dry gas bubbles through the tared bulbs *F*, containing a caustic potash solution of 1.27 specific gravity, which absorbs the carbonic acid, and the residual air, containing water from the potash solution, passes on through the guard-tube *F*, which absorbs the moisture, and escapes through *G* and the filter-pump. The gas should flow at the rate of 4 to 5 bubbles per second. When the bulb *B* is empty, heat the contents of the flask carefully, finally boiling the liquid slowly, to expel the carbonic acid. Air should be passed through the apparatus for a few minutes after boiling, to insure the removal of all the carbonic acid. Caps should be placed over the inlet and outlet tubes of *F* while making the weighings, to prevent the absorption of carbonic acid or moisture. When the operation is completed, place the bulbs and guard-tubes *F* in the balance-case, and after a few minutes weigh them. The increase in weight divided by the weight of material used and the quotient multiplied by 100 is the percentage of carbonic acid.

A similar apparatus may be fitted up, using an ordinary flask, with cork connections and an empty U tube, as recommended by Gladding, instead of the condenser *D*.

In the determination of carbonic acid with Schroetter's or similar apparatus, proceed as follows: The description refers to Fig. 134. Fill the tube on the left, to above the upper bulb, with concentrated sulphuric acid, and that on the right with dilute hydrochloric acid. Weigh the flask and contents, then introduce approximately 1.5 to 2 grams of the powdered limestone, by the opening at the left, and weigh again. Lift the stopper on the hydrochloric-acid tube, and open the stop-cock and admit a little acid. In the decomposition of the stone, the carbonic acid is set free and bubbles through the sulphuric acid, which retains any watery vapor that would otherwise pass off with the gas. Repeat this operation from time to time until no more carbonic acid is disengaged. Heat gently to expel the carbonic acid from the solution, cool, and weigh. After cooling and wiping the apparatus, it should be placed inside the balance-case a few minutes before weighing. The loss in weight is that of the carbonic acid set free. Divide this weight by that of the limestone used and multiply the quotient by 100 to obtain the percentage of carbonic acid.

The carbonic acid in the limestone used in sugar manufacture is almost entirely combined with calcium; a small portion is sometimes in combination with magnesium. Occasionally the stone contains a vein of dolomite, a carbonate of calcium and magnesium.

In the absence of gypsum, calcium sulphate, if either the percentages of calcium or magnesium and carbonic acid are given, the percentages of the two carbonates may be calculated: The percentage of calcium oxide ( $\text{CaO}$ )  $\times 1.7847$  = percentage of calcium carbonate ( $\text{CaCO}_3$ ); the percentage of carbonic acid in the magnesium carbonate ( $\text{MgCO}_3$ ) multiplied by 1.9164 = the percentage of magnesium carbonate.

*Example.*—A sample of limestone contains 54.8 per cent calcium oxide and 43.4 per cent carbonic acid; required, the percentages of calcium and magnesium carbonates.

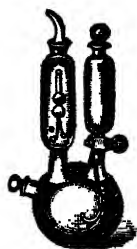


FIG. 134.  
Schroetter's  
Alkalimeter.

#### CALCULATION

$$\begin{aligned}
 54.8 \times 1.7875 &= 97.96, \text{ per cent calcium carbonate} \\
 97.96 - 54.8 &= 43.16, \text{ carbonic acid in the calcium carbonate} \\
 43.4 - 43.16 &= 0.24, \text{ carbonic acid in the magnesium carbonate} \\
 0.24 \times 1.916 &= 0.46, \text{ the per cent magnesium carbonate.}
 \end{aligned}$$

Many sugar-house chemists calculate the carbonates in this way, in order to economize time. In many cases this method will supply all the information necessary relative to the purity of the stone, but it is not usually advisable to depend entirely upon it. A serious objection to this process is the fact that there may be slight errors in the determinations of the calcium and carbonic acid which would lead to false deductions. It is advisable, as a rule, to determine both the bases and the acids.

**408. Determination of Sulphate.**—The limestone may contain small quantities of calcium sulphate. Digest 5 grams or more of the powdered limestone with hydrochloric acid, using heat. Dilute the solution, filter it, and wash the residue thoroughly with hot water. Concentrate the filtrate, in a beaker, to a volume of about 50 ml., heat it to boiling and add a solution of barium chloride, a few drops at a time, maintaining the boiling temperature. Remove the beaker from the lamp, after each addition of the chloride, to permit the barium sulphate to settle, and test the supernatant liquid for sulphate. Continue the boiling of the solution and the additions of the reagent so long as a precipitate forms; set aside overnight. Collect the precipitated barium sulphate in a tared Gooch crucible, wash it with hot water, heat it to redness, cool and weigh it. The weight of barium sulphate  $\times .343 \div$  weight of limestone used  $\times 100$  = percentage of sulphuric anhydride ( $\text{SO}_3$ ); the weight of barium sulphate  $\times .5832 \div$  weight of limestone used  $\times 100$  = percentage of calcium sulphate.

**409. Notes on the Analysis of Limestone.**—It may be necessary in some of the determinations to use a larger portion of the stone than 1 gram. If so, it is convenient to use a multiple of 1 gram, dissolve it and dilute the solution to a definite volume, 5 grams, to 500 ml. for example, and use measured portions of this solution for the determinations.

A Gooch crucible will usually be found much more convenient for the filtrations and ignitions than filter-paper and an ordinary crucible. Alundum may be substituted for the filter or Gooch, except in determining total silica.

In these methods of analysis, only those determinations are given which are necessary in judging a limestone for sugar-house purposes.

Sundstrom<sup>2</sup> has suggested a method for the rapid analysis of a limestone, an abstract of which follows:

(a) Weigh two portions of 1 gram each of the finely powdered sample, transfer them to small dishes and add about 100 ml. of distilled water to each. To one portion add 25 ml. of normal hydrochloric acid (444), cover the dish with a watch-glass until all action ceases; heat the solution to boiling, cool and titrate it with normal sodium hydrate (448), using methyl orange as an indicator. The number of milliliters of normal hydrochloric acid—the number of milliliters of normal soda solution = milliliters of normal hydrochloric acid required to react with the carbonates of lime and magnesia.

(b) To the second portion of 1 gram, cautiously add 5 milliliters of concentrated hydrochloric acid, keeping the dish covered to avoid loss. After all effervescence ceases, evaporate the material to complete dryness over a low flame. When dry, cool, take up with a little hot water and a few drops of hydrochloric acid; heat to boiling, filter through an ashless filter, washing all insoluble portions into the filter, and wash free of all traces of chlorides with boiling water.

(c) Dry the filter and contents; ignite in a tared platinum crucible to bright redness, cool under a desiccator and weigh for silica ( $\text{SiO}_2$ ).

(d) Neutralize the filtrate and washings from (b) with ammonium hydrate in slight excess; heat to boiling, collect the precipitate and wash free of

<sup>2</sup> Journal of the Society of Chemical Industry, 16, 520.

chlorides. Dry and ignite the filter and contents; cool and weigh for oxides of iron and aluminum ( $\text{Fe}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ ).

(e) Heat the filtrate and washings from (d) to boiling, add a concentrated solution of ammonium oxalate, also heated to boiling. Allow the mixture to stand until clear, which, if the analysis has been rightly conducted, requires two or three minutes; decant the clear solution into a filter, dissolve the precipitate in hydrochloric acid and reprecipitate with ammonium hydroxide. Allow to settle and decant as before, and then wash the whole precipitate into the filter and wash with hot water until free of chlorides and oxalates. Dry the filter and contents, ignite in a platinum crucible, at first cautiously, then over a blast-lamp, until the residue is converted into calcium oxide ( $\text{CaO}$ ); cool under a desiccator, weigh and calculate the weight to terms of calcium carbonate ( $\text{CaCO}_3$ ) as in the previous methods. Titrate the residue with normal hydrochloric acid as a check.

Divide the percentage of calcium carbonate by 5 (= milliliters of normal hydrochloric acid required for calcium carbonate), subtract the quotient from the number of milliliters of normal hydrochloric acid required for (a), and multiply the remainder by 4.2 to obtain the percentage of  $\text{MgCO}_3$ .

Sundstrom states that this method is very rapid and sufficiently accurate for technical purposes.

#### ANALYSIS OF LIME

**410. Determination of the Calcium Oxide in Lime.**—Add sufficient water (30 ml. ca.) to 10 grams of lime, in a mortar, to form a thick milk. Add an excess of pure sucrose in the form of a solution of 35–40° Brix and mix it intimately with the lime, which forms a soluble saccharate. Transfer the solution and residue to a 100-cc. flask, using a sugar solution of the above composition to wash the last portions from the mortar and to complete the volume to 100 ml.; mix and filter. Titrate 10 ml. of the filtrate with a normal solution of hydrochloric acid (444), using phenolphthalein or lacmoid as an indicator. The burette reading  $\times .028$  = the weight of calcium oxide ( $\text{CaO}$ ) in 1 gram of the lime, and  $\times 100$  = percentage of calcium oxide.

**411. Determination of the Proportion of Unburned and Slaked Lime.**—Slake 1 gram of lime with water, add an excess of normal sulphuric acid (446) and heat to expel carbonic acid if present; add a few drops of methyl orange solution or other suitable indicator, and ascertain the excess of sulphuric acid used, by titration with normal sodium hydrate (448). Calculation: (milliliters of normal sulphuric acid — milliliters of normal soda solution)  $\times .028$  = the total weight of calcium, as calcium oxide, in 1 gram of the lime, and  $\times 100$  = the percentage of total calcium as calcium oxide. This number — percentage of calcium oxide = percentage of unburned and slaked lime as calcium oxide.

**412. Determination of Calcium Oxide, etc. Degener-Lunge Method.**—Both the above determinations may be made with one titration using phenacetoline as suggested by Degener and applied by Lunge.

Slake a weighed portion of the lime with water, add a few drops of phenacetoline solution and titrate with normal hydrochloric acid. Add the acid

until the yellow color changes to a red, and read the burette. This reading multiplied by .028 gives the weight of calcium oxide. Continue the addition of the acid; the solution remains of a red color until all the calcium is saturated, then changes to a golden yellow. It is advisable to make this titration a few times for practice with material of known composition. The burette reading multiplied by .028 gives the total weight of calcium as calcium oxide. The unburned and slaked limes are determined by difference.

**413. Complete Analysis.**—The methods described for limestones may be applied for a further analysis of the lime if required.

#### ANALYSIS OF SULPHUR AND SULPHUROUS ACID

**414. Estimation of the Impurities.**—Transfer 0.5 gram of the powdered sulphur to a flask provided with a well-fitted glass stopper. Add at one time an excess of saturated bromine-water and shake thoroughly. Water dissolves 2 to 3.25 per cent of bromine at ordinary temperatures, and, as at least 15 parts bromine are required for 1 part of sulphur, it is advisable to use from 275 to 400 ml. of the bromine water to insure sufficient of the reagent for the oxidation of the sulphur to sulphuric acid. Boil the solution to expel the excess of bromine, collect the residue and wash with hot water; dry and weigh. A Gooch crucible is convenient for collecting the residue. The weight of the residue  $\times 200$  = percentage of impurities. The percentage of sulphur may be calculated directly from that of the sulphuric acid in the filtrate (408), or with sufficient accuracy for practical purposes, by subtracting the percentage of impurities from 100.

Commercial roll-sulphur is usually very pure. Its quality can generally be satisfactorily determined from its color and relative freedom from dust and small fragments.

**415. Estimation of Sulphuric Acid in Sulphurous Acids.**—The following method is recommended by Herzfeld : <sup>3</sup> Pass the sulphurous acid gas into a 40 to 50 per cent sucrose solution for ten to twenty minutes. Test a portion of the solution for sulphuric acid by the addition of barium chloride and hydrochloric acid. An insoluble precipitate of barium sulphate forms in the presence of sulphuric acid. If a quantitative test is desired, a measured volume of the gas must be used and the precipitated sulphate must be collected in a Gooch filter and be washed with hot water, dried and ignited to redness. The weight of the barium sulphate  $\times 0.343$  = the weight of sulphur trioxide ( $\text{SO}_3$ ) in the volume of gas used.

<sup>3</sup> Zeitschrift, 1911, 917; Int. Sugar Journ., 1912, 14, 113.



## CHAPTER XXXVIII

### ANALYSES OF FLUE-GASES

**416. Remarks on Flue-gases.**—The analysis of flue-gases is made as a part of the boiler-house control for the purpose of ascertaining whether the full efficiency of the fuel is being obtained. Occasionally factories using the carbonation process use the flue-gases as a source of carbon dioxide.

For these purposes the ordinary volumetric analysis is usually employed and the constituents are reported in volume per cents. The constituents determined are carbonic acid ( $\text{CO}_2$ ), carbonic oxide ( $\text{CO}$ ), oxygen ( $\text{O}$ ), and nitrogen ( $\text{N}$ ).

**417. Preparation of the Apparatus and Reagents.**—The most convenient apparatus for this analysis is that of Orsat, Fig. 135. A form of this apparatus having a four-way stopcock instead of those on the pipettes *B*, *C*, *D* and *G* is often used, but that illustrated is easier to manipulate and is preferable.

The apparatus consists of a water-jacketed burette *A*, for the measurement of the gases and three absorption U tubes *B*, *C*, *D*, with suitable connections and stopcocks. The absorption branches of the U tubes are filled with pieces of small-bore glass tubing to increase the surface exposed to the gases.

The absorption-tubes are connected with the burette by barometer tubing of very small bore. The branches of the U tubes at the rear are usually connected with a soft rubber bulb (not shown in the figure), to prevent exposing the solutions to the air and thus weakening them. The water-jacket on the burette is for maintaining a fairly constant temperature during the test.

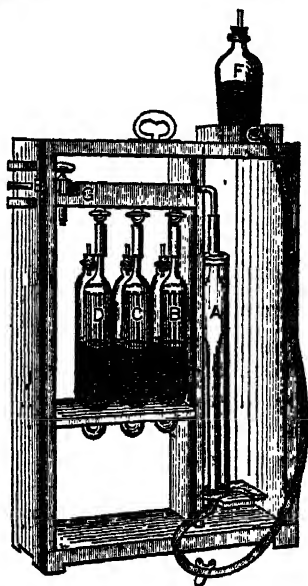


FIG. 135.—Orsat's Gas Apparatus.

The following solutions are used in the absorption-tubes:

*For tube B:* Use a concentrated solution of caustic potash ( $\text{KOH}$ ) of about 60° Brix.

*For tube C:* Dissolve 5 parts of pyrogallie acid in 50 parts of hot water and add 100 parts of caustic potash solution of approximately 50° Brix.

*For tube D:* This tube is to be filled with a solution of cuprous chloride prepared as follows: Place 35 grams of cupric chloride in a bottle, add 200 ml. of concentrated hydrochloric acid and a quantity of copper turnings or copper foil. Stopper the bottle and set it aside for two days, shaking it occasionally, than add 120 ml. water.

Ready prepared cuprous chloride may be obtained of the dealers, and used, instead of making it in the laboratory. Twenty-six grams of cuprous chloride should be dissolved in 200 ml. concentrated HCl as above, 120 ml. of water added, and copper trimmings or foil placed in the storage bottle. Each of the tubes in *D* should have a piece of copper wire in it.

The U tube should be filled half full of the solutions and the rubber bulb then connected with the branches, or in lieu of the bulb, oil may be poured on the surfaces of the liquids. The bulb is preferable, however.

**418. Sampling and Analysis of Flue-gases.**—A piece of half-inch iron pipe should be inserted into each flue, leading to the chimney, reaching about half-way to the center of the flue. A double-acting rubber-bulb pump, with suitable valves, is used in drawing the gas from the flue and discharging it into a soft rubber bulb used as a receiver. The bulb should be filled with the gases and emptied several times, to expel all the air it or the pump may contain, and then be filled with the sample and its rubber tube closed with a pinch-cock. It is convenient to have a number of these bulbs so that duplicate samples may be drawn from each flue. A pair of aspirator bottles may also be used for collecting the sample of gas.

In analyzing the samples proceed as follows: Fill the bottle *F*, Fig. 135, with distilled water (which has previously been saturated with the gases to be analyzed) close the cocks on *BC* and *D* and open the 3-way cock *G* to the air; lift *F* until the water fills the burette to the upper mark, then close the 3-way cock and pinch-cock on the rubber tube connecting *F* with the burette, and place *F* on the table.

A U tube containing a little water and a light plug of cotton in each branch is connected at *E*.

Open the pinch-cock and then cautiously open the cock on the U tube *B* and let the caustic-potash solution rise to the mark on the upper part of the tube, lowering *F* still more if the pressure is not sufficient. Fill the tubes *C* and *D* in the same way, filling the burette with water each time by altering the position of the bottle *F*. With the absorption branch of each U tube filled with its solution and the burette with water, the apparatus is ready for the tests. Connect the sample-bulb with the small U tube on *E* and permit a little of the gas to escape into the air through the side branch of the 3-way cock *G*, to expel the air or previous sample from the connections.

Open the cock to *G* to connect with the apparatus, and the pinch-cock on the water-tube and let the gas displace the water in the burette. Hold the bottle *F*, so that the level of the water in it will be level with the zero of the burette, disconnect the sample-bulb and manipulate the cock *G* so that the levels of the water in the bottle and burette will be the same. Lift the bottle *F* and cautiously open the cock on *B*, and let the gas displace the caustic potash, filling and emptying the U tube with gas by manipulating the bottle, but at

no time letting the water rise above the 100 mark on the burette. The caustic potash solution will absorb the carbonic acid. As soon as absorption ceases, hold the bottle with the surface of the water in it at the level of that in the burette and note the burette reading, which is the percentage of carbonic acid ( $\text{CO}_2$ ). Next repeat these manipulations with the residue of the gas using U tube *C*. Note the total reading of the burette and subtract the first reading from it to obtain the percentage of oxygen ( $\text{O}$ ). Again proceed with the last residue of gas as before, using U tube *D*. The second burette reading subtracted from the third gives the percentage of carbon monoxide ( $\text{CO}$ ). The

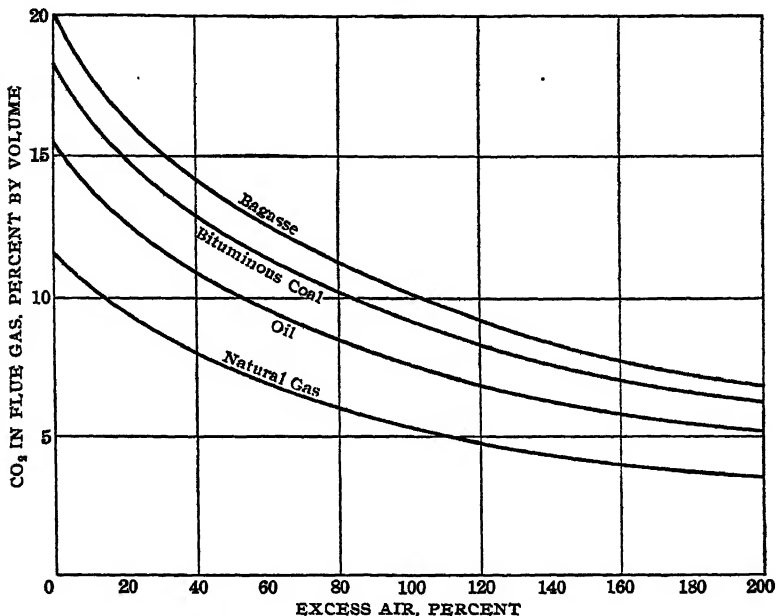


FIG. 136.—Excess Air and  $\text{CO}_2$  for Various Fuels.

final residue usually consists almost entirely of nitrogen ( $\text{N}$ ) and the percentage is obtained by subtracting the third burette reading from 100. The gases may contain very small quantities of sulphuretted hydrogen and sulphurous acid; these introduce a slight error in the determinations.

Sulphuretted hydrogen is tested for with filter-paper moistened with lead acetate or subacetate, which turns black in the presence of the gas.

Sulphurous acid may be detected by shaking a little of the gas in a test-tube with iodized starch solution. If this acid is present the blue color is discharged.

After each analysis the residual gas should be expelled from the apparatus and the burette left filled with water. The cocks should be well greased with vaseline or some preparation for this purpose.

**419. Automatic CO<sub>2</sub> Devices.**—Many modern boiler-houses are equipped with automatic devices for analyzing the flue-gases. These generally indicate and record at short intervals the percentage of CO<sub>2</sub> present. They are of two general types, direct and indirect, the direct actually analyzing the gas sample by absorbing the CO<sub>2</sub> in caustic as in the Orsat, while the indirect depend upon the difference in density of carbon dioxide and air.

These devices all require close attention to keep them in accurate working order. Descriptions and detailed directions can be obtained from the makers.

**420. Interpretation of Flue Gas Analyses.**—The burning of any fuel under boiler conditions requires the use of an excess of air; *i.e.*, more air than the theoretical amount required to supply oxygen to all the combustible. Any attempt to limit the air to within a close approximation of the theoretical will result in smoke, loss of unburned carbon in the form of soot and incomplete combustion as shown by the presence of carbon monoxide (CO) in the flue gases. The loss under these conditions may be very great as only about one-third the heat value is obtained when carbon is burned to the monoxide as when it is completely burned to carbon dioxide. There should be no carbon monoxide whatever in the flue gases.

On the other hand too great an excess of air also means an avoidable loss. The amount of air is indicated by the percentage of CO<sub>2</sub> in the flue gases, the relationship varying with the fuel as shown in Fig. 136. It is the heat required to raise this excess air from outside temperatures to that of the stack gases that causes the loss. As has been shown in Fig. 19 in Chapter IV there is a difference of about 15 per cent of the bagasse as fired between the loss due to 200 per cent excess air and 100 per cent excess air.

## CHAPTER XXXIX

### FERMENTATION AND MICROORGANISMS IN SUGAR MANUFACTURE

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In cane sugar manufacture fermentation is an ever-present problem from the time that the juice is first extracted until the sugar reaches its final state of purity and is distributed as the refined granulated product. While the susceptibility of the intermediate products of raw sugar manufacture to deteriorate decreases from the raw juice to the finished sugar, this fact does not constitute a criterion of the relative importance of the problem at the different stages of the manufacturing process, since the finished products are subjected to these changes over much longer periods than any of the products of the preceding stages. Cane juices as freshly extracted<sup>1</sup> contain a very large number of micro-organisms, which gain access to it from the rind of the cane and from the trash and dirt adhering thereto, and also from other more extraneous sources. This number may vary from less than one hundred thousand to several million per cubic centimeter, depending upon the cleanliness of the cane, and the sanitary condition of the mill in which the extraction is effected. Where the rind of the cane is uninjured and where special methods of extraction are used so as to eliminate all chances of outside infection, cane juices may be obtained in a practically sterile condition. Freshly extracted cane juices possess a considerable degree of germicidal action,<sup>2</sup> and for several hours after their extraction the number of microorganisms decrease as a result of enzymic action. In frozen or burnt cane this protective action is lost, and as a result the deterioration of the juice of such cane progresses with undiminished rapidity from the time that it is extracted until the action is checked either by outside intervention or by the exhaustion of the food supply for the microorganisms.

**421. Classification of Microorganisms.**—The microorganisms occurring in cane juices may be classified according to their economic importance in sugar manufacture, as follows:

*Bacteria.*—(1) Species which destroy sucrose without the formation of gum, and which are of importance only in the juice.

<sup>1</sup> W. L. Owen, La. Expt. Station, Bulletin No. 125.

<sup>2</sup> Browne, C. A. J. Am. Chem. Society, Vol. XXVIII, 1906.

(2) Gum-forming species which include the dextran forming *Leuconostoc mesenteroides*, and the levan-forming *mesentericus* group of bacteria. The former is of importance only in the juices and the dilute liquors while the other group persist throughout the entire manufacturing process, and may cause a slow inversion of sucrose in sirups and molasses.

(3) Species known as thermophiles, which develop at temperatures between 46°–73° and are sometimes responsible for the deterioration of hot clarified juices, and often develop in the filter presses and juice settlers. Among the best known of these species is the *Microspira Northii*.<sup>3</sup>

**Mold Fungi.**—(1) Very strongly inverting species which are active in juices as well as in sirups, and constitute the most active agency in the deterioration of raw sugars, example, *Aspergillus repens*,<sup>4</sup> and *Aspergillus niger*.

(2) Species which induce only a feeble inversion of sucrose at the density corresponding to that of the molasses films surrounding sugar crystals, but which may induce considerable losses of sucrose in cane juices—example, the *Citromyces* group, and *Monilia fusca* (Browne).

(3) Species which are very prevalent in sugar products, but are of little economic significance owing to their weak inverting power—example, *Monilia nigra* (Browne).<sup>5</sup>

**Yeast.**—(1) Those species which are found in raw juices, and which induce an active inversion of sucrose at that density.

(2) Species of non-sporulating yeast known as *Torulae*, containing no invertase, and which induce an active destruction of reducing sugar, and often a selective action upon levulose, in cane sirups and molasses, and in the molasses films surrounding raw sugar crystals.

**422. Factors Affecting the Number of Microorganisms in Cane Juices.**—The number and kind of microorganisms occurring in cane juices depend to a very large extent upon the amount and nature of the substances adhering to the rind of the cane, from which the extracted juice becomes infected during milling. The mere mechanical filtration of freshly extracted cane juices has been found to eliminate approximately 75 per cent on an average of the microorganisms originally present, while the use of vegetable carbons and Filter-Cel<sup>6</sup> in the proportions in which they are ordinarily used in sugar manufacture removes approximately 99 per cent of the microorganisms originally contained in the juice. It is evident from these results that it is of very great importance to use the most efficient type of juice strainers in order to eliminate at the very beginning as many as possible of the microorganisms with which the freshly extracted juice is contaminated.

**423. Mill Sanitation.**—From the fact that freshly extracted cane juices are immediately infected with a very large number of microorganisms, many of which are capable of rapidly inverting sucrose, it is highly important to avoid exposing them to the deteriorative action of the latter for more than

<sup>3</sup> Carpenter, C. A. and Bomonti, H. F. Rep. Hawaiian Sugar Technologists, 1921.

<sup>4</sup> Owen, W. L. Facts About Sugar, 1925.

<sup>5</sup> Browne, C. A. Ind. and Eng. Chem., Vol. 10, 1918.

<sup>6</sup> Owen, W. L. Int. Sugar J., 26, 1924.

the briefest possible interval. It is also highly essential to keep the mill as free as possible from deteriorated material such as fermented portions of bagasse or particles of trash which have absorbed fermented juice. Frequent washing and cleaning of mills, and the use of steam at frequent intervals, has been known to reduce greatly the drop in purity between crusher and mill juices.<sup>7</sup> It still is a debatable question whether the use of antiseptics at the mills is to be recommended as a regular procedure, owing to the large quantities that would be required and the expense that would be involved. However, the use of suitable germicides from time to time in connection with the washing down and the cleaning of the mills, or after any undue delay due to breakdowns or other causes is to be recommended. Formaldehyde in the proportion of 1 : 200<sup>8</sup> or chloride of lime in the proportion of 1 : 50 will prove a very efficient method of maintaining the sanitary conditions of mills. This is, of course, a much higher concentration than would ever be required for the preservation of juices, for which purpose formalin in the proportion of from 1 : 5000 to 1 : 10,000 is usually recommended (see Chapter X). For the elimination of infection from fermented juices, especially of the viscous type, chloride of lime will be found to be the more efficient of the two germicides, owing to its greater penetrating power.

424. *The Influence of Temperature upon the Nature of the Fermentation of Cane Juices.*—Although the nature of the infection of the juice from sound cane at the time of its extraction is subject to comparatively little variation, the type of the predominant fermentation varies considerably at different times. One of the most prominent factors determining the course of the spontaneous fermentation of cane juice is the temperature to which it happens to be exposed. Experiments have shown<sup>9</sup> that at temperatures below 20° C. the viscous type of fermentation seems to predominate more frequently than at temperatures between 20° C. and 30° C. This would tend to explain the susceptibility of juices from frozen cane to dextran fermentation.

The temperature limits of two of the most important groups of gum-forming bacteria found in cane juices are as follows:

Group	Min. Temp.	Op. Temp.	Max. Temp.
<i>Leuconostoc mesenteroides</i> ...	11-14	30-35	40-43
<i>Bacillus mesentericus</i> (Levan forming bacteria).....	15-22	37	45

Other factors which tend to determine the type of fermentation which cane juices undergo are (1) pH of the juices and (2) the amount and kind of contaminating material that gains access to it.

<sup>7</sup> McCleery, W. L. Rept. Hawaiian Sugar Technologists, 1925.

<sup>8</sup> Owen, W. L. La. Expt. Station, Bulletin No. 153.

<sup>9</sup> Owen, W. L. Int. Sugar J., 17, 1921.

**425. Number of Microorganisms at the Various Stages of Raw Sugar Manufacture.**—In the process of sugar manufacture the microorganisms occurring in the raw juice tend to be successively eliminated at all of the subsequent stages of the manufacturing process, by the methods of clarification and by the temperatures employed in connection therewith. The following data illustrate the eliminative effects of clarification upon the number of microorganisms occurring at the different stages of raw sugar manufacture: <sup>10, 11, 12</sup>

Product	Number Microorganisms per Gram or Cc.
Raw juice.....	280,000
Sulphured juice.....	35,000
Limed juice.....	37,500
Defecated juice.....	750
Sirup.....	400
Masseccuite.....	450
Sugars.....	600
Molasses.....	35,000
Wash waters.....	25,000
Filter-press cake.....	1,500,000

It is evident from the above data that good clarification of the juices in raw sugar manufacture will be reflected in a lower microbial content of the various products at the successive stages of manufacture.

**426. Other Types of Fermentation in Sugar Cane Products.**—The viscous fermentation of cane juices is associated with a very pronounced reducing action, resulting in the marked bleached appearance of the fermented juice. Various deoxidation products result from this fermentation, among which Browne <sup>13</sup> found mannite to be the most commonly occurring. The amount of mannite occurring in fermenting juices, however, is subject to wide variations, owing to the readiness with which it may be utilized as a source of energy for other groups of microorganisms.

In addition to the viscous fermentation of cane juices, and the ordinary alcoholic fermentation, there are other less commonly occurring, and less well understood fermentative phenomenon, of which Browne has made a very thorough study. Among these may be mentioned the Cellulosic fermentation, in which sucrose is assimilated by certain forms of bacteria such as *Bacterium xylinum*, with the formation of cellulose. This organism forms clumps of gelatinous sheaths in cane juices and Browne found that the product of this

<sup>10</sup> Owen, W. L. La. Expt. Station, Bulletin No. 125.

<sup>11</sup> Church, M. B. Sugar, 1921.

<sup>12</sup> Kopeloff, N. and L. La. Planter, 1919.

<sup>13</sup> Browne, C. A. Jour. Am. Chem. Soc., Vol. 28 (1906).



fermentation consisted of cellulose, and represented approximately 7 per cent of the total sugars fermented.

Among the interesting products of fermentation in sugar cane products may be mentioned mannan, which Browne identified in the deposits and scums formed in fermenting juices, sirups and molasses, and also chitine, which he isolated from the scums which form on the surface of molasses left over in the hot-room. These scums consist of the growth of the fungus *Citromyces*, and Browne's investigations have shown that the former are characterized by an exceptionally high fat content (27.5 per cent) which in composition very strikingly resembles butter fat.

## CHAPTER XL

### .SPECIAL REAGENTS

**427. Litmus Solution.**—This solution may be prepared either (a) directly from pure azolitmin which may be purchased from the dealers, or (b, c) by separating this coloring matter from crude litmus. The crude litmus should not be used without purification of the azolitmin in the preparation of either the solution or the test-paper, since the other coloring matters present impair the sensibility.

(a) Dissolve 1 gram of pure azolitmin in 100 ml. of dilute alcohol containing about 20 ml. of 85 per cent alcohol, by weight. Filter the solution and preserve it as indicated in (b).

(b) Boil 100 grams of commercial litmus with 600 ml. of distilled water and set it aside to cool and deposit the residue. Decant the clear solution and evaporate it on the water-bath to about 200 ml. Filter the concentrate and dilute the filtrate to 300 ml. with distilled water and add 100 ml. of diluted sulphuric acid containing 16.2 grams of the pure concentrated acid. Heat the mixture four hours on the water-bath with frequent stirring. Collect the precipitate which forms and wash it with cold water, until the washings assume a peculiar fiery red color and on addition of caustic alkali a deep blue and not a violet color. Reject the washings and dissolve out the purified coloring-matter with 100 ml. lukewarm 90 per cent alcohol to which a few drops of ammonia have been added. Distill off the alcohol, after filtration, and evaporate the residue to dryness on the water-bath. Dissolve the dried residue in 600 ml. of distilled water and neutralize the solution with sodium hydrate. The neutral solution should have a pure violet tint.

Litmus solution decomposes when stored in a stoppered bottle. It should be kept in a salt-mouth bottle, which should be only half filled with the solution, and a loose plug of cotton should be placed in the mouth of the bottle to keep out dust and admit air.

(c) Exhaust powdered litmus with successive portions of hot alcohol to remove the coloring-matter that accompanies the azolitmin. Digest the residue in cold water acidulated with sulphuric or hydrochloric and reject the solution. Treat the residue with boiling water to extract the azolitmin and then filter the solution. The filtrate should be neutralized and preserved as in (b).

**428. Litmus Papers.**—These papers are prepared by soaking filter-paper in acid (red), neutral and alkaline (blue) azolitmin solution. The paper should

be of the best quality and be thoroughly washed. Well-washed quantitative filter-paper is suitable. A paper is sometimes used to which the solution is only applied to one side, the other being left white for comparisons.

The neutral paper is prepared by soaking the paper in the azolitmin solution obtained by one of the methods of the previous paragraph. The sheet of paper should be drawn over a glass rod to remove the surplus solution, and then hung to dry in a room free of laboratory fumes. The sheet should be occasionally turned when first hung to prevent the solution from accumulating at the edges by drainage.

The red paper is prepared as above, but from an azolitmin solution that has been faintly acidulated with sulphuric. The acidulation is conveniently and efficiently accomplished as follows: Divide the azolitmin solution into two equal parts and faintly acidulate one and then mix the two. Repeat this proceeding until the mixture is very faintly red. Test the sensitiveness of the solution by soaking a strip of filter-paper in it. The paper should be slightly red when dried. Proceed as above in preparing an alkali solution for the blue paper, except using a dilute sodium hydrate solution.

**429. Turmeric or Curcuma Paper.**—Digest powdered commercial turmeric or curcumin in water to remove all water-soluble coloring-matter. This repeated digestion is best conducted by decantation. Treat the residue with alcohol to dissolve the curcumin. Neutralize the solution with dilute caustic soda solution and soak filter-paper in it. On account of the usual acidity of paper, the solution may even be slightly alkaline. Dry the paper by suspending the sheet from a line with clips and then again soak it in the curcumin solution, this having in the meantime been again neutralized. Again dry the paper and repeat these operations until an exceedingly sensitive paper is obtained. This paper turns a reddish brown with alkali and yellow with acid. The neutral paper just verges on a brown tint. This paper is usually sufficiently sensitive for all factory purposes.

Turmeric paper was formerly extensively used in the control of the ordinary defecation process. The paper is turned a reddish brown by lime and this color may easily be seen by artificial light. Cane-juice that has very slight turmeric alkalinity in the cold usually becomes neutral on heating, due to the combination of the lime with the organic acids at the higher temperature.

**430. Phenolphthalein Solution.**—Dissolve 1 gram of phenolphthalein in 100 ml. of diluted alcohol and neutralize it with acid or alkali as may be necessary.

**431. Phenolphthalein or Dupont Paper.**—This paper is made by soaking the very finest quality of filter- or glazed paper in an alcoholic solution of phenolphthalein. Dupont regulated the sensitiveness of the paper by adding dilute sulphuric acid to the alcoholic solution and made the sensitiveness correspond to different proportions of lime or other alkalinity of juices. This paper is much used in the control of sulphitation processes.

**432. Corallin or Rosalic Acid Solution.**—A solution of commercial corallin in 90 per cent alcohol, nearly neutralized, may be used.

For determining the alkalinity or acidity of molasses (305) the alcohol-soluble corallin used as a stain in microscopy is recommended.

**433. Methyl Orange.**—Dissolve 1 gram of the pure material in distilled water and make to 1 liter.

**434. Phenacetolin Solution.**—Dissolve 2 grams of the reagent in 1000 ml. of strong alcohol and neutralize the solution.

**435. Iodate Paper.**—Dissolve 2 grams of starch in 100 ml. of water with heating and add 0.2 gram of iodate of potassium, dissolved in 5-ml. of water. Soak filter-paper in this solution and dry it.

This paper is used in testing for sulphurous acid, which, even in slight traces, frees the iodine and colors the paper blue through the reaction with the starch.

**436. Nessler's Solution.**—Dissolve 62.5 grams of potassium iodide in 250 ml. of water. Set aside about 10 ml. of this solution; add to the larger portion a solution of mercuric chloride until the precipitate formed no longer redissolves. Add the 10 ml. of potassium iodide solution; then continue the addition of mercuric chloride very cautiously until only a light permanent precipitate forms. Dissolve 150 grams of caustic potash in 150 ml. water, cool and add it gradually to the above solution. Dilute the mixture to 1 liter.

**437. Subacetate of Lead.**—Heat to boiling, for about half an hour, 860 grams of neutral lead acetate, 260 grams of litharge, and two liters of water. Cool, settle, and decant the clear solution.

This solution may be prepared without heat, provided the mixture is set aside several hours, with frequent shaking. The solution should be diluted with cold, recently boiled, distilled water to 54.3° Brix. (1.25 sp. gr.)

**438. Neutral Lead Acetate.**—Dissolve neutral lead acetate in distilled water to make a saturated solution.

**439. Hydrate of Alumina. "Alumina Cream."**—To a saturated solution of common alum in water add ammonia little by little until in slight excess, then enough additional alum to render the solution slightly acid to litmus paper. The sulphates may remain in solution to precipitate the excess of lead.

When precipitation of the lead is not wished, the hydrate of alumina should be washed by decantation with water until it contains only traces of sulphates.

**440. Preparation of Pure Sugar.**—The following method of purifying sugar, for use in testing polariscopes, was adopted by the Fourth International Congress of Applied Chemistry, Paris, 1900, on the recommendation of the committee appointed with a view to unifying the methods of sugar analysis used in various countries: Prepare a hot saturated solution of the purest commercial sugar obtainable, and precipitate the sugar with absolute ethyl alcohol. Spin the precipitated sugar in the laboratory centrifugal and wash it with alcohol. Redissolve and reprecipitate the sugar as before, washing it in the centrifugal with alcohol. The sugar so obtained should be dried between pieces of blotting-paper and preserved in a stoppered jar. The moisture in the sugar should be determined and proper allowance made for it when weighing the sample for analysis.

The preparation of pure sucrose is a difficult operation as will be found by reference to the work of Bates and Jackson, pages 94 to 99 of Circular No. 44 of the Bureau of Standards. For most sugar laboratory work any dry high-grade granulated sugar (preferably cube sugar) will be as close to pure sucrose

as is obtainable by any ordinary method and this may generally be employed wherever pure sucrose is specified for use.

**441. Preparation of Pure Invert Sugar.**—Dissolve 4.75 grams of pure sucrose in 75 ml. of water, add 10 ml. of dilute hydrochloric acid (24.85 Brix at 20° C. as is used in Clerget methods) and set aside for twenty-four hours at a temperature not lower than 20° C. Neutralize the acid carefully with sodium hydroxide, shaking constantly during the addition of the alkali. Make to 1 liter. Ten ml. of this solution contains 0.050 gram of invert sugar.

**442. Fehling's Solution, Soxhlet's Modification.**—Two solutions are employed which are mixed in equal portions immediately before use. All the glucose methods described in this book use this reagent.

(a) *Copper Sulfate Solution.*—Dissolve 34.639 grams of copper sulphate ( $\text{CuSO} \cdot 5\text{H}_2\text{O}$ ) in water, dilute to 500 ml. and filter through prepared asbestos.

(b) *Alkaline Tartrate Solution.*—Dissolve 173 grams of sodium hydroxide in water, dilute to 500 ml., allow to stand for two days and filter through prepared asbestos.

**443. Normal Acid and Alkali Solutions.**—According to Sutton <sup>1</sup> "Normal solutions, as a rule, are so prepared that 1 liter shall contain the hydrogen equivalent of the active reagent weighed in grams ( $\text{H} = 1.008$ ).<sup>1</sup>" Thus normal sulphuric acid contains 49.040 grams  $\text{H}_2\text{SO}_4$  per liter; normal hydrochloric acid, 36.465 grams  $\text{HCl}$  per liter, etc. Half-normal, one-fifth normal, and one-tenth normal (decinormal) solutions are frequently used, and may be prepared by diluting the normal solutions. Normal, half-normal one-fifth normal solutions, etc., are usually indicated as follows:  $\text{N}$ ,  $\frac{\text{N}}{2}$ ,  $\frac{\text{N}}{5}$ ,  $\frac{\text{N}}{10}$  etc.

These solutions are prepared and checked as described in the following sections.

**444. Standard Hydrochloric Acid.**—The reagent acid usually has a specific gravity of 1.20, approximately. Acid of this specific gravity contains about 40 per cent of hydrochloric acid, hence a little less than 50 grams of it are required to contain the 18.233 grams necessary to form a half-normal solution which is a convenient strength. It is advisable to dilute a somewhat larger quantity of the acid, e.g., 40 ml. to 1000 ml., with distilled water, rather than to attempt to approximate the correct quantity.

To standardize this acid, first prepare pure sodium carbonate as follows: Wash several grams of any good brand of ordinary "baking soda" several times with cold water by decantation to remove possible soluble chlorides and sulphates; dry in a hot air oven at 100° C. and then heat to dull redness in a platinum dish for half an hour (or to constant weight). The heating changes the sodium bicarbonate,  $\text{NaHCO}_3$ , to sodium carbonate,  $\text{Na}_2\text{CO}_3$ . Store the pure  $\text{Na}_2\text{CO}_3$  in a desiccator in a glass-stoppered bottle. To standardize the acid, weigh accurately about 1.1 grams of the carbonate, dissolve in 100 ml. of distilled water, add 2-3 drops methyl orange and titrate to the point where the color changes from yellow to pinkish-orange. One gram of pure  $\text{Na}_2\text{CO}_3 = 37.736$  ml. of  $\text{N}/2$  acid. Weight of  $\text{Na}_2\text{CO}_3 \times 37.736 =$  ml. acid that should have been used if acid is exactly  $\text{N}/2$ . This figure divided into milliliters of

<sup>1</sup> "Volumetric Analysis"

acid actually used  $\times 1000$  = number of milliliters of the acid that must be made up with water to 1 liter to yield exactly  $N/2$  acid. The preliminary titration should show the acid too strong in order that it may be diluted to the correct strength.

For example: Suppose 1.11 gram sodium carbonate takes 39.70 ml. of acid to neutralize. Then

$$1.11 \times 37.736 = 41.89$$

$$\frac{39.70 \times 1000}{41.89} = 947.7$$

Then 947.7 ml. of the acid must be made to 1 liter to obtain exactly  $N/2$  acid.

After the dilution the acid should again be titrated against sodium carbonate in the same manner as above to check the correctness of the dilution. The acid so checked will serve as a convenient basis for the preparation of other standard acids and alkalies.

$$\begin{aligned} 1 \text{ ml. normal hydrochloric acid} &= .036465 \text{ gram HCl} \\ &= .035457 \text{ gram Cl} \\ &= .028035 \text{ gram CaO} \end{aligned}$$

**445. Standard Oxalic Acid.**—This is the simplest of the normal solutions to prepare, and when strictly pure oxalic acid can be obtained it may be used in checking the preparation of all the standard alkali and acid solutions. Oxalic acid of tested purity as obtained from the dealers will serve the purpose.

Reject all crystals that show indications of efflorescence. Dissolve 63.024 grams of this acid in distilled water and dilute to 1000 ml. to prepare the normal solution, or, preferably, dry the powdered acid at  $100^{\circ}$  C. to constant weight and use 45.018 grams in preparing the normal solution. It is advisable to employ weaker solutions than the normal, usually the one-tenth normal acid. This should be prepared from the normal solution as required, since the latter keeps well, provided it is not exposed to direct sunlight.

$$1 \text{ ml. normal oxalic acid} = .06303 \text{ gram H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}.$$

**446. Standard Sulphuric Acid.**—Add approximately 28 ml. of concentrated sulphuric acid to distilled water, cool the solution, and dilute to 1000 ml. Standardize by titration with normal alkali or gravimetrically by precipitation with barium chloride. Many chemists use this method as the starting point in the preparation of standard acids and alkalies.

$$\begin{aligned} 1 \text{ ml. normal sulphuric acid} &= .049040 \text{ gram H}_2\text{SO}_4 \\ &= .028035 \text{ gram CaO} \\ &= .051815 \text{ gram SrO} \\ &= .076685 \text{ gram BaO} \end{aligned}$$

**447. Standard Sulphuric Acid for the Control of the Carbonation.**—Add approximately 21 ml. of concentrated sulphuric acid to distilled water, cool the solution, and dilute to 1000 ml. Titrate this solution with a normal soda or potash solution, using phenolphthalein as an indicator. Dilute the acid so that 14 ml. of it will be required to neutralize 10 ml. of the normal alkali.

$$\begin{aligned} 1 \text{ ml. this standard acid} &= .035 \text{ gram } \text{H}_2\text{SO}_4 \\ &= .02 \text{ gram } \text{CaO} \end{aligned}$$

It is usual to add the phenolphthalein to this solution before dilution to 1000 ml.

**448. Standard Alkali Solutions.**—Ammonium hydroxide ( $\text{NH}_4\text{OH}$ ), caustic soda ( $\text{NaOH}$ ), and caustic potash ( $\text{KOH}$ ) are used in preparing the alkali solutions. The normal soda or potash solutions are used, but the ammonia should be weaker, preferably decinormal, or, for Sidersky's method for reducing sugars, half-normal.

Dissolve 42 grams of chemically pure caustic soda in water, in preparing the normal reagent, cool the solution, dilute to 1000 ml. and standardize it by titration against a normal acid. In preparing the potash solution, use 58 grams of chemically pure caustic potash. Standard ammonia is prepared by diluting the reagent to approximately the required strength, and standardizing it by titration with decinormal or half-normal acid, as may be required, using cochineal as an indicator.

1 cc. normal caustic soda solution	= .04001 gram $\text{NaOH}$
	= .03100 gram $\text{Na}_2\text{O}$
1 cc. normal caustic potash solution	= .05610 gram $\text{KOH}$
	= .04710 gram $\text{K}_2\text{O}$
1 cc. half-normal ammonia solution	= .00852 gram $\text{NH}_3$
	= .01752 gram $\text{NH}_4\text{OH}$
1 cc. decinormal ammonia solution	= .00170 gram $\text{NH}_3$
	= .00351 gram $\text{NH}_4\text{OH}$

Phenolphthalein cannot be used as an indicator with ammonia.

**449. Decinormal Potassium Permanganate.**—Dissolve 3.16 grams of chemically pure, dry permanganate of potassium ( $\text{KMnO}_4$ ) in distilled water, and dilute to 1000 ml. This solution is conveniently checked by titration with decinormal oxalic acid. To 10 ml. of decinormal oxalic acid add several volumes of water and a few cubic centimeters of dilute sulphuric acid. Warm the solution to approximately  $60^\circ \text{C}$ ., and add the permanganate solution little by little. Discontinue the addition of the permanganate as soon as the solution acquires a faint pink or rose color. The temperature of the solution must be maintained at approximately  $60^\circ \text{C}$ ., and a little time must be allowed for the reaction.

Permanganate of potassium solution should be preserved in a tightly stoppered bottle, and should be checked from time to time. The formation of

a precipitate indicates a change in the solution. It is simpler to determine a factor from time to time, rather than attempt to maintain the solution strictly decinormal.

1 cc. decinormal permanganate of potash  $\left\{ \begin{array}{l} = 00316 \text{ gram KMnO}_4 \\ = 00636 \text{ gram Cu} \end{array} \right.$



# REFERENCE TABLES

## FOR USE IN

## SUGAR LABORATORIES

TABLE 1

SHOWING THE IMPURITIES PRESENT IN COMMERCIAL REAGENTS; ALSO THE  
STRENGTH OF SOLUTIONS, ETC., RECOMMENDED IN ANALYSIS

Name	Symbol	Impurities	Strength of Solution, etc.
Sulphuric Acid (Oil of Vitriol)	$H_2SO_4$	Pb, As, Fe, Ca, $HNO_3$ , $N_2O_4$	Concentrated and dilute. To dilute pour 1 part acid by measure into 9 parts distilled water. Use por- celain dish.
Nitric Acid	$HNO_3$	$H_2SO_4$ , HCl	Concentrated and dilute. To dilute add 1 part acid to 9 parts water.
Hydrochloric Acid (Muriatic Acid)	HCl	Cl, $Fe_2Cl_6$ , $H_2SO_4$ , $SO_2$ , As	Concentrated and dilute. Dilute = 1 part acid to 9 parts water.
Nitro-hydro- chloric Acid (Aqua regia)			Prepare when required by adding 3 parts hydro- chloric to 1 part nitric acid. Use concentrated acids.
Acetic Acid	$HC_2H_3O_2$	$H_2SO_4$ , HCl, Cu, Pb, Fe, Ca	Concentrated and dilute. Dilute = 1 part pure gla- cial acetic acid to 1 part water.
Sulphurous Acid	$H_2SO_3$		To charcoal, in a flask, add concentrated $H_2SO_4$ . Boil, wash the gas gener- ated by passing it through water, and finally pass it into very cold water. Pre- serve the solution in tightly- stoppered bottles.
Oxalic Acid	$H_2C_2O_4$	Fe, K, Na, Ca	Dissolve 1 part of crystal- lized acid in 9 parts dis- tilled water.
Hydrogen Sulphide	$H_2S$		Use in gaseous state or in water solution. Wash the gas.
Sodium Hydroxide or Potassium Hydroxide	NaOH, KOH	Al, $SiO_2$ , phos- phates, sulphates, and chlorides	Dissolve the stick soda or potash in 20 parts water. (Soda is less expensive, and will usually answer for most purposes in place of potash.)
Ammonium Hydroxide	$NH_4OH$	Sulphate, chlo- ride, carbonate, tarry matters	Stronger water of am- monia (.96 specific gravity) and $\frac{1}{2}$ above strength.

TABLE 1.—REAGENTS—*Continued*

Name	Symbol	Impurities	Strength of Solution, etc.
Barium Hydroxide	$\text{Ba}(\text{OH})_2$		Dissolve 1 part of the crystals in 20 parts water; filter, and preserve in stoppered bottle.
Calcium Hydroxide	$\text{Ca}(\text{OH})_2$		Slake lime in water, filter off the solution, and preserve out of contact with the air.
Sodium Ammonium Acid Phosphate (Microcosmic Salt)	$\text{Na}(\text{NH}_4)\text{HPO}_4$		Dry and powder the salt. It may be made as follows: Dissolve 7 parts sodium acid phosphate ( $\text{Na}_2\text{HPO}_4$ ) and 1 part ammonium chloride in 2 parts boiling water, filter, and separate the required salt by crystallization. Purify by recrystallization.
Sodium Tetraborate	$\text{Na}_2\text{B}_4\text{O}_7$		Heat to expel water of crystallization and powder.
Sodium Carbonate	$\text{Na}_2\text{CO}_3$	Chlorides, phosphates, sulphates, silicates.	Use the powdered salt or dissolve in 5 parts water.
Ammonium Sulphate	$(\text{NH}_4)_2\text{SO}_4$		Dissolve 1 part in 5 parts water.
Ammonium Chloride	$\text{NH}_4\text{Cl}$	Fe. Purify the commercial salt by the addition of ammonia; filter. Neutralize filtrate with $\text{HCl}$ ; concentrate and recrystallize.	Dissolve 1 part in 5 parts water.
Ammonium Nitrate	$\text{NH}_4\text{NO}_3$		Saturated solution.
Ammonium Oxalate	$(\text{NH}_4)_2\text{C}_2\text{O}_4$	Purify by recrystallization.	Dissolve 1 part in 20 parts water.
Ammonium Carbonate	$(\text{NH}_4)_2\text{CO}_3$	Pb, Fe, sulphates, chlorides.	Dissolve 1 part in 4 parts water, and add 1 part ammonia, specific gravity .880.
Ammonium Molybdate			Dissolve the salt in strong ammonia, decant the clear solution slowly into strong nitric acid, stirring thoroughly till the precipitate redissolves.
Ammonium Sulphide	$(\text{NH}_4)_2\text{S}$		Saturate 3 parts ammonia with $\text{H}_2\text{S}$ , then add 2 parts ammonia.
Ammonium Poly-sulphide	$(\text{NH}_4)_2\text{S}_x$		Prepared by dissolving sulphur in ammonium sulphide.
Potassium Sulphate	$\text{K}_2\text{SO}_4$		Dissolve 1 part in 10 parts water.
Potassium Iodide	KI	Iodate, carbonate.	Dissolve 1 part in 50 parts water.

TABLE 1—REAGENTS—*Continued*

Name	Symbol	Impurities	Strength of Solution, etc.
Potassium Chromate	$K_2CrO_4$	Sulphates	Dissolve 1 part in 10 parts water.
Potassium Bichromate	$K_2Cr_2O_7$		Dissolve 1 part in 10 parts water.
Potassium Ferricyanide	$K_3Fe(CN)_6$		Dissolve 1 part in 12 parts water. Better to prepare solution when required.
Potassium Ferrocyanide	$K_4Fe(CN)_6$		Dissolve 1 part in 12 parts water, or, for glucose work, 1 part in 50 parts water.
Barium Chloride	$BaCl_2$	Purify the commercial salt by passing $H_2S$ through it and crystallizing.	Dissolve 1 part in 10 parts water.
Barium Nitrate	$Ba(NO_3)_2$		Dissolve 1 part in 15 parts water.
Barium Carbonate	$BaCO_3$		Add water to the powdered carbonate and preserve in salt-mouthed bottle.
Calcium Chloride	$CaCl_2$	Fe	Dissolve 1 part in 5 parts water.
Calcium Sulphate	$CaSO_4$		Dissolve as much of the salt as possible in water (in the cold), filter, and preserve the filtrate.
Magnesium Sulphate	$MgSO_4$	Fe, Ni, etc.	Dissolve 1 part in 10 parts water.
Ferrous Sulphate	$FeSO_4$		Dissolve 1 part in 10 parts cold water.
Ferric Chloride	$FeCl_3$		Dissolve 1 part in 10 parts water.
Cobaltous Nitrate	$Co(NO_3)_2$		Dissolve 1 part in 10 parts water.
Cupric Sulphate	$CuSO_4$	Fe, Zn	For sugar work purify by repeated crystallizations unless very high purity salt is obtainable. For Fehling solution see 442. For ordinary work dissolve 1 part in 10 parts water.
Mercuric Chloride	$HgCl_2$		Dissolve 1 part in 20 parts water.
Mercurous Nitrate	$HgNO_3$		Dissolve 1 part in 20 parts water acidulated with 1.2 part nitric acid. Filter into a bottle containing a little metallic mercury.
Platinic Chloride	$PtCl_4$		Dissolve 1 part in 10 parts water.
Argentive Nitrate	$AgNO_3$		Dissolve 1 part in 10 parts water.
Stannous Chloride	$SnCl_2$		Dissolve pure tin in strong HCl in the presence of platinum. Dilute with 4 volumes dilute HCl. Keep granulated tin in the bottle.

TABLE 2

INTERNATIONAL ATOMIC WEIGHTS (PARTIAL LIST) FOR 1925

On the Basis of Oxygen = 16

(Jour. Am. Chem. Soc., Vol. 47 (1925), p. 597)

Name	Symbol	Weight	Name	Symbol	Weight
Aluminum.....	Al	26.97	Magnesium.....	Mg	24.32
Antimony.....	Sb	121.77	Manganese.....	Mn	54.93
Arsenic.....	As	74.96	Mercury.....	Hg	200.61
Barium.....	Ba	137.37	Molybdenum.....	Mo	96.0
Bismuth.....	Bi	209.00	Nickel.....	Ni	58.69
Boron.....	B	10.82	Nitrogen.....	N	14.008
Bromine.....	Br	79.916	Oxygen.....	O	16
Cadmium.....	Cd	112.41	Phosphorus.....	P	31.027
Calcium.....	Ca	40.07	Platinum.....	Pt	195.23
Carbon.....	C	12.000	Potassium.....	K	39.096
Chlorine.....	Cl	35.457	Radium.....	Ra	225.95
Chromium.....	Cr	52.01	Selenium.....	Se	79.2
Cobalt.....	Co	58.94	Silicon.....	Si	28.06
Copper.....	Cu	63.57	Silver.....	Ag	107.880
Fluorine.....	F	19.00	Sodium.....	Na	22.997
Gold.....	Au	197.2	Strontium.....	Sr	87.63
Hydrogen.....	H	1.008	Sulphur.....	S	32.064
Iodine.....	I	126.932	Tin.....	Sn	118.70
Iron.....	Fe	55.84	Uranium.....	U	238.17
Lead.....	Pb	207.20	Zinc.....	Zn	65.38

TABLE 3

EQUIVALENTS OF THE UNITED STATES CUSTOMARY  
AND METRIC WEIGHTS AND MEASURES

## Miscellaneous Equivalents

1 Avoirdupois pound	=	453.5924277 grams
1 Meter	=	39.37 inches (U. S. law of 1866)
1 U. S. mile	=	1.60935 kilometers
1 Kilometer	=	0.62137 U. S. mile
1 Acre	=	0.4047 hectare
1 Hectare	=	2.471 acres
1 U. S. liquid ounce	=	29.574 cubic centimeters
1 U. S. apothecaries' dram	=	3.6967 cubic centimeters
1 U. S. dry quart	=	1.1012 liters
1 U. S. bushel	=	0.35239 hectoliter
1 Long ton (2240 av. lbs.)	=	1016.05 kilograms
1 Short ton (2000 av. lbs.)	=	907.18 kilograms
1 Metric ton	=	2204.62 avoirdupois pounds

\* From "Tables of Equivalents," 4th Ed., U. S. Bureau of Standards.

TABLE 3—TABLE OF EQUIVALENTS—Continued

LENGTHS				AREAS					
Inches	Millimeters	Inches	Centimeters	Feet	Meters	Square Inches	Square Millimeters	Square Feet	Square Meters
0.03937 = 1		0.3937 = 1		1	= 0.304801	0.00155 =	1	1	= 0.09290
0.07874 = 2		0.7874 = 2		2	= 0.609601	0.00310 =	2	2	= 0.18581
0.11811 = 3		1 = 2.54001		3	= 0.914402	0.00465 =	3	3	= 0.27871
0.15748 = 4		1.1811 = 3		3.28083 = 1		0.00620 =	4	4	= 0.37161
0.19685 = 5		1.5748 = 4		4	= 1.219202	0.00775 =	5	5	= 0.46452
0.23622 = 6		1.9685 = 5		5	= 1.524003	0.00930 =	6	6	= 0.55742
0.27559 = 7		2 = 5.08001		6	= 1.828804	0.01085 =	7	7	= 0.55032
0.31496 = 8		2.3622 = 6		6.56167 = 2		0.01240 =	8	8	= 0.74323
0.35433 = 9		2.7559 = 7		7	= 2.133604	0.01395 =	9	9	= 0.83613
1 = 25.4001		3 = 7.62002		8	= 2.438405	1	= 645.16	10.764 = 1	
2 = 50.8001		3.1496 = 8		9	= 2.743205	2	= 1,290.33	21.528 = 2	
3 = 76.2002		3.5433 = 9		9.84250 = 3		3	= 1,935.49	32.292 = 3	
4 = 101.6002		4 = 10.16002		13.12333 = 4		4	= 2,580.65	43.055 = 4	
5 = 127.0003		5 = 12.70003		16.40417 = 5		5	= 3,225.81	53.819 = 5	
6 = 152.4003		6 = 15.24003		19.68500 = 6		6	= 3,870.98	64.583 = 6	
7 = 177.8004		7 = 17.78004		22.96583 = 7		7	= 4,516.14	75.347 = 7	
8 = 203.2004		8 = 20.32004		26.24667 = 8		8	= 5,161.30	86.111 = 8	
9 = 228.6005		9 = 22.86005		29.52750 = 9		9	= 5,806.46	96.875 = 9	

TABLE 3—TABLES OF EQUIVALENTS—Continued

VOLUMES				CAPACITIES		MASSES		
Cubic Inches	Cubic Cent.	Cubic Feet	Cubic Meters	U. S. Liquid Gallons	Liters	Grains	Grams	Avoldupois Pounds
0.0610 = 1	1	1	= 0.02832	0.26417 = 1	1	1	= 0.06480	1 = 0.45359
0.1220 = 2	2	2	= 0.05663	0.52834 = 2	2	2	= 0.12960	2 = 0.90718
0.1831 = 3	3	3	= 0.08495	0.79251 = 3	3	3	= 0.19440	2.20462 = 1
0.2441 = 4	4	4	= 0.11327	1	= 3.78543	4	= 0.25920	3 = 1.36078
0.3051 = 5	5	5	= 0.14159	1.05668 = 4	4	5	= 0.32399	4 = 1.81437
0.3661 = 6	6	6	= 0.16990	1.32085 = 5	5	6	= 0.38879	4.40924 = 2
0.4272 = 7	7	7	= 0.19822	1.58502 = 6	6	7	= 0.45359	5 = 2.26796
0.4882 = 8	8	8	= 0.22654	1.84919 = 7	7	8	= 0.51839	6 = 2.72155
0.5492 = 9	9	9	= 0.25485	2	= 7.57087	9	= 0.58319	6.61387 = 3
1 = 16.3872	1	35.314 = 1		2.11336 = 8	8	15.4324 = 1		7 = 3.17515
2 = 32.7743	2	70.629 = 2		2.37753 = 9	9	30.8647 = 2		8 = 3.62874
3 = 49.1615	3	105.943 = 3		3	= 11.35630	46.2971 = 3		8.81849 = 4
4 = 65.5486	4	141.258 = 4		4	= 15.14174	61.7294 = 4		9 = 4.08233
5 = 81.9358	5	176.572 = 5		5	= 18.92717	77.1618 = 5		11.02311 = 5
6 = 98.3230	6	211.887 = 6		6	= 22.71261	92.5941 = 6		13.22773 = 6
7 = 114.7101	7	247.201 = 7		7	= 26.49804	108.0265 = 7		15.43236 = 7
8 = 131.0873	8	282.516 = 8		8	= 30.28348	123.4589 = 8		17.63698 = 8
9 = 147.4845	9	317.830 = 9		9	= 34.06891	138.8912 = 9		19.84160 = 9

TABLE 4

## MENSURATION

**Parallelogram:** Area of any parallelogram = base  $\times$  altitude; area of rhombus = product of two adjacent sides  $\times$  sine of angle included between them.

**Trapezium:** Area = (diagonal  $\times$  sum of perpendicular let fall on it from opposite angles)  $\div 2$  = Area of the two triangles into which it may be divided.

**Trapezoid:** Area = half the sum of the two parallel sides  $\times$  the perpendicular distance between them.

**Any Quadrilateral:** Divide the quadrilateral into two triangles and find the sum of the areas of these, or area = half the product of the two diagonals  $\times$  the sine of the angle at their intersection.

**Triangle:** Area = base  $\times$  half the altitude = half the product of two sides  $\times$  the sine of the included angle = half the sum of the three sides minus each side severally; multiply this half sum and the three remainders together and extract the square root of the product. Area of an equilateral triangle = one fourth the square of one of its sides  $\times 0.433013$ .

Hypotenuse and one side of a right-angle triangle being given to find the other side: Required side =  $\sqrt{\text{Hypotenuse}^2 - \text{given side}^2}$ ; if the two sides are equal side = hypotenuse  $\times 0.7071$ .

Area given to find base: Base =  $2 \times \text{area} \div \text{perpendicular height}$ .

Area given to find height: Height =  $2 \times \text{area} \div \text{base}$ .

Two sides and base given to find perpendicular height, both angles at base are acute: Base: sum of the sides: difference of the sides : difference of the divisions of the base made by drawing the perpendicular. Half this difference added to or subtracted from half the base will give the divisions of it.

**Polygon:** Area of irregular polygon; draw diagonals dividing the polygon into triangles and find the sum of the areas of these.

Area of regular polygon: Area = (length of a side  $\times$  perpendicular distance of side to center  $\times$  number of sides)  $\div 2$  = half the perimeter  $\times$  perpendicular distance of side to center.

Perpendicular to center = half of one side  $\times$  cotangent of the angle subtended by half the side.

**Irregular Figure (Laboratory method):** Draw the figure on paper of uniform thickness and cut it out; compare the weight of this piece with that of a sheet of the paper of known area.

**Circle:** Ratio of circumference to diameter = 3.1415929 (usually taken as 3.1416).

This number is usually represented by the Greek letter  $\pi$ .

Circumference = diameter  $\times \pi$ .

Length of an arc: 360 : number of degrees of the arc :: circumference of the circle : length of the arc, or diameter of the circle  $\times$  number of degrees in the arc  $\times 0.0087266$  = length of the arc.

Area of a circle = square of the radius  $\times \pi$  = square of diameter  $\times 0.7854$ .

Areas of circles are to each other as the squares of their diameters.

**Ellipse:** Area = product of the semi-axes  $\times 3.1416$  = product of the axes  $\times 0.7854$ .

**Prism:** Area = (perimeter of base  $\times$  altitude)  $\div 2$  = areas of the two ends.

Volume = area of base  $\times$  altitude.

**Pyramid:** Convex surface of a regular pyramid = perimeter of base  $\times$  half the slant height.

Volume = area of base  $\times$  one third of altitude.

Area of a frustum of a regular pyramid = Half the slant height  $\times$  sum of the perimeters of the two bases (= convex surface) + area of the two bases.

Volume of a frustum of a regular pyramid = sum of areas of the two bases and a mean proportional between them  $\times$  one third of the altitude. (Mean proportional between two numbers = square root of their product.)

TABLE 4—MENSURATION—Continued

**Rectangular Prismoid** (a solid bounded by six planes, of which two bases are rectangles, having corresponding sides parallel, and the four upright sides are trapezoids):  
 Volume = (sum of the areas of the bases + four times the area of a parallel section equidistant from the bases)  $\times$  one sixth of the altitude.

**Cylinder:** Area of convex surface = circumference of base  $\times$  altitude. Convex surface + the two end areas = total area.

Volume of a cylinder = area of base  $\times$  altitude.

**Cone:** Total area = circumference of base  $\times$  half slant height (=convex surface) + area of the base.

Volume of a cone = area of base  $\times$  one third of altitude.

Frustum of a cone: Area = (half the side  $\times$  sum of the circumference of the two bases) + area of the two bases.

Volume of frustum of a cone = (area of two bases + a mean proportional between them  $\times$  one third of the altitude). (See Volume of a frustum of a pyramid relative to a mean proportional.)

**Parabola:** Area = base  $\times \frac{2}{3}$  altitude.

**Sphere:** Surface = diameter  $\times$  circumference of its great circle = square of diameter  $\times 3.1416$  = convex surface of its circumscribing cylinder.

Surfaces of spheres are to each other as the squares of their diameters.

Volume of a sphere = surface  $\times$  one third of the radius = cube of the diameter  $\times 0.5236$ .

Volumes of spheres are to each other as the cubes of their diameters.

**Cask:** Volume of a cask in U. S. gallons =  $(39 \times \text{square of bung diameter} + 25 \times \text{square of head diameter} + 26 \times \text{the product of the diameters}) \times \text{length} \div 26,470$ .

**Molasses Tanks, Crystallizers:** See Table 7.



TABLE 5

## CIRCUMFERENCES AND AREAS OF CIRCLES

Diam- eter, Inches	Circum- ference, Inches	Area, Square Inches	Diam- eter, Inches	Circum- ference, Inches	Area, Square Inches	Diam- eter, Inches	Circum- ference, Inches	Area, Square Inches
$\frac{1}{8}$	.39270	.012272	7	21.991	38.4846	13	40.841	132.733
$\frac{1}{4}$	.78540	.049087	$\frac{1}{8}$	22.384	39.8713	$\frac{1}{8}$	41.233	135.297
$\frac{3}{8}$	1.1781	.110447	$\frac{1}{4}$	22.776	41.2826	$\frac{1}{4}$	41.626	137.887
$\frac{1}{2}$	1.5708	.19635	$\frac{3}{8}$	23.169	42.7184	$\frac{3}{8}$	42.019	140.501
$\frac{5}{8}$	1.9635	.306796	$\frac{1}{2}$	23.562	44.1787	$\frac{1}{2}$	42.412	143.139
$\frac{3}{4}$	2.3562	.441787	$\frac{5}{8}$	23.955	45.6636	$\frac{5}{8}$	42.804	145.802
$\frac{7}{8}$	2.7489	.601322	$\frac{3}{4}$	24.347	47.1731	$\frac{3}{4}$	43.197	148.49
1	3.1416	.7854	$\frac{7}{8}$	24.740	48.7071	$\frac{7}{8}$	43.590	151.202
$\frac{1}{8}$	3.5343	.99402	8	25.133	50.2656	14	43.982	153.938
$\frac{1}{4}$	3.9270	1.2272	$\frac{1}{8}$	25.525	51.8487	$\frac{1}{8}$	44.375	156.7
$\frac{3}{8}$	4.3197	1.4849	$\frac{1}{4}$	25.918	53.4593	$\frac{1}{4}$	44.768	159.485
$\frac{1}{2}$	4.7124	1.7671	$\frac{3}{8}$	26.311	55.0884	$\frac{3}{8}$	45.160	162.296
$\frac{5}{8}$	5.1051	2.0739	$\frac{1}{2}$	26.704	56.7451	$\frac{1}{2}$	45.553	165.13
$\frac{3}{4}$	5.4978	2.4053	$\frac{5}{8}$	27.096	58.4264	$\frac{5}{8}$	45.946	167.99
$\frac{7}{8}$	5.8905	2.7612	$\frac{3}{4}$	27.489	60.1322	$\frac{3}{4}$	46.338	170.874
2	6.2832	3.1416	$\frac{7}{8}$	27.882	61.8625	$\frac{7}{8}$	46.731	173.782
$\frac{1}{8}$	6.6759	3.5466	9	28.274	63.6174	15	47.124	176.715
$\frac{1}{4}$	7.0686	3.9761	$\frac{1}{8}$	28.667	65.3968	$\frac{1}{8}$	47.517	179.673
$\frac{3}{8}$	7.4613	4.4301	$\frac{1}{4}$	29.060	67.2008	$\frac{1}{4}$	47.909	182.655
$\frac{1}{2}$	7.8540	4.9087	$\frac{3}{8}$	29.452	69.0293	$\frac{3}{8}$	48.302	185.661
$\frac{5}{8}$	8.2467	5.4119	$\frac{1}{2}$	29.845	70.8823	$\frac{1}{2}$	48.695	188.692
$\frac{3}{4}$	8.6394	5.9396	$\frac{5}{8}$	30.238	72.7599	$\frac{5}{8}$	49.087	191.748
$\frac{7}{8}$	9.0321	6.4918	$\frac{3}{4}$	30.631	74.6621	$\frac{3}{4}$	49.480	194.828
3	9.4248	7.0686	$\frac{7}{8}$	31.023	76.5888	$\frac{7}{8}$	49.873	197.933
$\frac{1}{8}$	9.8175	7.6699	10	31.416	78.54	16	50.265	201.062
$\frac{1}{4}$	10.210	8.2958	$\frac{1}{8}$	31.809	80.5158	$\frac{1}{8}$	50.658	204.216
$\frac{3}{8}$	10.603	8.9462	$\frac{1}{4}$	32.201	82.5161	$\frac{1}{4}$	51.051	207.395
$\frac{1}{2}$	10.996	9.6211	$\frac{3}{8}$	32.594	84.5409	$\frac{3}{8}$	51.444	210.593
$\frac{5}{8}$	11.388	10.3206	$\frac{1}{2}$	32.987	86.5903	$\frac{1}{2}$	51.836	213.825
$\frac{3}{4}$	11.781	11.0447	$\frac{5}{8}$	33.379	88.6643	$\frac{5}{8}$	52.229	217.077
$\frac{7}{8}$	12.174	11.7933	$\frac{3}{4}$	33.772	90.7628	$\frac{3}{4}$	52.622	220.354
4	12.566	12.5664	$\frac{7}{8}$	34.165	92.8858	$\frac{7}{8}$	53.014	223.655
$\frac{1}{8}$	12.959	13.3641	11	34.558	95.0334	17	53.407	226.981
$\frac{1}{4}$	13.352	14.1863	$\frac{1}{8}$	34.950	97.2055	$\frac{1}{8}$	53.800	230.331
$\frac{3}{8}$	13.744	15.033	$\frac{1}{4}$	35.343	99.4022	$\frac{1}{4}$	54.192	233.706
$\frac{1}{2}$	14.137	15.9043	$\frac{3}{8}$	35.736	101.6234	$\frac{3}{8}$	54.585	237.105
$\frac{5}{8}$	14.530	16.8002	$\frac{1}{2}$	36.128	103.8691	$\frac{1}{2}$	54.978	240.529
$\frac{3}{4}$	14.923	17.7206	$\frac{5}{8}$	36.521	106.1394	$\frac{5}{8}$	55.371	243.977
$\frac{7}{8}$	15.315	18.6655	$\frac{3}{4}$	36.914	108.4343	$\frac{3}{4}$	55.763	247.45
5	15.708	19.635	$\frac{7}{8}$	37.306	110.7537	$\frac{7}{8}$	56.156	250.948
$\frac{1}{8}$	16.101	20.629	12	37.699	113.098	18	56.549	254.47
$\frac{1}{4}$	16.493	21.6476	$\frac{1}{8}$	38.092	115.466	$\frac{1}{8}$	56.941	258.016
$\frac{3}{8}$	16.886	22.6907	$\frac{1}{4}$	38.485	117.859	$\frac{1}{4}$	57.334	261.587
$\frac{1}{2}$	17.279	23.7583	$\frac{3}{8}$	38.877	120.277	$\frac{3}{8}$	57.727	265.183
$\frac{5}{8}$	17.671	24.8505	$\frac{1}{2}$	39.270	122.719	$\frac{1}{2}$	58.119	268.803
$\frac{3}{4}$	18.064	25.9673	$\frac{5}{8}$	39.663	125.185	$\frac{5}{8}$	58.512	272.448
$\frac{7}{8}$	18.457	27.1086	$\frac{3}{4}$	40.055	127.677	$\frac{3}{4}$	58.905	276.117
6	18.850	28.2744	$\frac{7}{8}$	40.448	130.192	$\frac{7}{8}$	59.298	279.811
$\frac{1}{8}$	19.242	29.4648	13	39.841	132.733	19	59.690	283.529
$\frac{1}{4}$	19.635	30.6797	$\frac{1}{8}$	40.234	135.297	$\frac{1}{8}$	60.083	287.272
$\frac{3}{8}$	20.028	31.9191	$\frac{1}{4}$	40.626	137.887	$\frac{1}{4}$	60.476	291.04
$\frac{1}{2}$	20.420	33.1831	$\frac{3}{8}$	41.019	140.501			
$\frac{5}{8}$	20.813	34.4717	$\frac{1}{2}$	41.412	143.139			
$\frac{3}{4}$	21.206	35.7848	$\frac{5}{8}$	41.804	145.802			
$\frac{7}{8}$	21.598	37.1224	$\frac{3}{4}$	42.197	148.49			

TABLE 6  
TEMPERATURE CONVERSION TABLES  
By ALBERT SAUVEUR  
(Courtesy of Chem. and Met. Eng.)

0 to 100				100 to 1000			
C.	F.	C.	F.	C.	F.	C.	F.
-17.8	0 32	10.0	50 122.0	38	100 212	260	500 932
-17.2	1 33.8	10.6	51 123.8	43	110 230	266	510 950
-16.7	2 35.6	11.1	52 125.6	49	120 248	271	520 968
-16.1	3 37.4	11.7	53 127.4	54	130 266	277	530 986
-15.6	4 39.2	12.2	54 129.2	60	140 284	282	540 1004
-15.0	5 41.0	12.8	55 131.0	66	150 302	288	550 1022
-14.4	6 42.8	13.3	56 132.8	71	160 320	293	560 1040
-13.9	7 44.6	13.9	57 134.6	77	170 338	299	570 1058
-13.3	8 46.4	14.4	58 136.4	82	180 356	304	580 1076
-12.8	9 48.2	15.0	59 138.2	88	190 374	310	590 1094
-12.2	10 50.0	15.6	60 140.0	93	200 392	316	600 1112
-11.7	11 51.8	16.1	61 141.8	99	210 410	321	610 1130
-11.1	12 53.6	16.7	62 143.6	100	212 413	327	620 1148
-10.6	13 55.4	17.2	63 145.4	104	220 428	332	630 1166
-10.0	14 57.2	17.8	64 147.2	110	230 446	338	640 1184
- 9.44	15 59.0	18.3	65 149.0	116	240 464	343	650 1202
- 8.89	16 60.8	18.9	66 150.8	121	250 482	349	660 1220
- 8.33	17 62.6	19.4	67 152.6	127	260 500	354	670 1238
- 7.78	18 64.4	20.0	68 154.4	132	270 518	360	680 1256
- 7.22	19 66.2	20.6	69 156.2	138	280 536	366	690 1274
- 6.67	20 68.0	21.1	70 158.0	143	290 554	371	700 1292
- 6.11	21 69.8	21.7	71 159.8	149	300 572	377	710 1310
- 5.56	22 71.6	22.2	72 161.6	154	310 590	382	720 1328
- 5.00	23 73.4	22.8	73 163.4	160	320 608	388	730 1346
- 4.44	24 75.2	23.3	74 165.2	166	330 626	393	740 1364
- 3.89	25 77.0	23.9	75 167.0	171	340 644	399	750 1382
- 3.33	26 78.8	24.4	76 168.8	177	350 662	404	760 1400
- 2.78	27 80.6	25.0	77 170.6	182	360 680	410	770 1418
- 2.22	28 82.4	25.6	78 172.4	188	370 698	416	780 1436
- 1.67	29 84.2	26.1	79 174.2	193	380 716	421	790 1454
- 1.11	30 86.0	26.7	80 176.0	199	390 734	427	800 1472
- 0.56	31 87.8	27.2	81 177.8	204	400 752	432	810 1490
0	32 89.6	27.8	82 179.6	210	410 770	438	820 1508
0.56	33 91.4	28.3	83 181.4	216	420 788	443	830 1526
1.11	34 93.2	28.9	84 183.2	221	430 806	449	840 1544
1.67	35 95.0	29.4	85 185.0	227	440 824	454	850 1562
2.22	36 96.8	30.0	86 186.8	232	450 842	460	860 1580
2.78	37 98.6	30.6	87 188.6	238	460 860	466	870 1598
3.33	38 100.4	31.1	88 190.4	243	470 878	471	880 1616
3.89	39 102.2	31.7	89 192.2	249	480 896	477	890 1634
4.44	40 104.0	32.2	90 194.0	254	490 914	482	900 1652
5.00	41 105.8	32.8	91 195.8			488	910 1670
5.56	42 107.6	33.3	92 197.6			493	920 1688
6.11	43 109.4	33.9	93 199.4			499	930 1706
6.67	44 111.2	34.4	94 201.2			504	940 1724
7.22	45 113.0	35.0	95 203.0			510	950 1742
7.78	46 114.8	35.6	96 204.8			516	960 1760
8.33	47 116.6	36.1	97 206.6			521	970 1778
8.89	48 118.4	36.7	98 208.4			527	980 1796
9.44	49 120.2	37.2	99 210.2			532	990 1814
		37.8	100 212.0			538	1000 1832

NOTE.—The numbers in bold face type refer to the temperature either in degrees Centigrade or Fahrenheit which it is desired to convert into the other scale. If converting from degrees Fahrenheit to degrees Centigrade the equivalent temperature will be found in the left column, while if converting from degrees Centigrade to degrees Fahrenheit, the answer will be found in the column on the right.

TABLE 7

FOR THE CALCULATION OF THE WANTAGE (EMPTY SPACE) IN HORIZONTAL CYLINDRICAL TANKS (TANK CARS, CRYSTALLIZERS, ETC.), IN U. S. GALLONS AND CUBIC FEET

(Let  $W$  = wantage;  $D$  = diameter of tank;  $I$  = depth of empty space ("inches out");  $L$  = mean length of tank;  $F$  = area of segment,  $W = (D^2 \times F \times L) \div 231$ , wantage in U. S. gallons, or dividing by 1728 gives wantage in cubic feet. All dimensions are in inches.) See page 368.

$I+D$	$F$	$I+D$	$F$	$I+D$	$F$	$I+D$	$F$	$I+D$	$F$
.001	.00004	.051	.01512	.101	.04148	.151	.07459	.201	.11262
.002	.00012	.052	.01556	.102	.04208	.152	.07531	.202	.11343
.003	.00022	.053	.01601	.103	.04269	.153	.07603	.203	.11423
.004	.00034	.054	.01646	.104	.04310	.154	.07675	.204	.11503
.005	.00047	.055	.01691	.105	.04391	.155	.07747	.205	.11584
.006	.00062	.056	.01737	.106	.04452	.156	.07820	.206	.11665
.007	.00078	.057	.01783	.107	.04514	.157	.07892	.207	.11746
.008	.00095	.058	.01830	.108	.04575	.158	.07965	.208	.11827
.009	.00113	.059	.01877	.109	.04638	.159	.08038	.209	.11908
.010	.00133	.060	.01924	.110	.04700	.160	.08111	.210	.11990
.011	.00153	.061	.01972	.111	.04763	.161	.08185	.211	.12071
.012	.00175	.062	.02020	.112	.04826	.162	.08258	.212	.12153
.013	.00197	.063	.02068	.113	.04889	.163	.08332	.213	.12235
.014	.00220	.064	.02117	.114	.04953	.164	.08406	.214	.12317
.015	.00244	.065	.02165	.115	.05016	.165	.08480	.215	.12399
.016	.00268	.066	.02215	.116	.05080	.166	.08554	.216	.12481
.017	.00294	.067	.02265	.117	.05145	.167	.08629	.217	.12563
.018	.00320	.068	.02315	.118	.05209	.168	.08704	.218	.12646
.019	.00347	.069	.02366	.119	.05274	.169	.08779	.219	.12728
.020	.00375	.070	.02417	.120	.05338	.170	.08853	.220	.12811
.021	.00403	.071	.02468	.121	.05404	.171	.08929	.221	.12894
.022	.00432	.072	.02519	.122	.05469	.172	.09004	.222	.12977
.023	.00462	.073	.02571	.123	.05534	.173	.09080	.223	.13060
.024	.00492	.074	.02624	.124	.05600	.174	.09155	.224	.13144
.025	.00523	.075	.02676	.125	.05666	.175	.09231	.225	.13227
.026	.00555	.076	.02729	.126	.05733	.176	.09307	.226	.13311
.027	.00587	.077	.02782	.127	.05799	.177	.09384	.227	.13394
.028	.00619	.078	.02835	.128	.05866	.178	.09460	.228	.13478
.029	.00653	.079	.02889	.129	.05933	.179	.09537	.229	.13562
.030	.00686	.080	.02943	.130	.06000	.180	.09613	.230	.13646
.031	.00721	.081	.02997	.131	.06067	.181	.09690	.231	.13731
.032	.00756	.082	.03052	.132	.06135	.182	.09767	.232	.13815
.033	.00791	.083	.03107	.133	.06203	.183	.09845	.233	.13900
.034	.00827	.084	.03162	.134	.06271	.184	.09922	.234	.13984
.035	.00864	.085	.03218	.135	.06339	.185	.10000	.235	.14069
.036	.00901	.086	.03274	.136	.06407	.186	.10077	.236	.14154
.037	.00938	.087	.03330	.137	.06476	.187	.10155	.237	.14239
.038	.00976	.088	.03387	.138	.06545	.188	.10233	.238	.14324
.039	.01015	.089	.03444	.139	.06614	.189	.10312	.239	.14409
.040	.01054	.090	.03501	.140	.06683	.190	.10390	.240	.14494
.041	.01093	.091	.03558	.141	.06753	.191	.10468	.241	.14580
.042	.01133	.092	.03616	.142	.06822	.192	.10547	.242	.14665
.043	.01173	.093	.03674	.143	.06892	.193	.10626	.243	.14751
.044	.01214	.094	.03732	.144	.06962	.194	.10705	.244	.14837
.045	.01255	.095	.03790	.145	.07033	.195	.10784	.245	.14923
.046	.01297	.096	.03849	.146	.07103	.196	.10864	.246	.15009
.047	.01339	.097	.03908	.147	.07174	.197	.10943	.247	.15095
.048	.01382	.098	.03968	.148	.07245	.198	.11023	.248	.15182
.049	.01425	.099	.04027	.149	.07316	.199	.11102	.249	.15268
.050	.01468	.100	.04087	.150	.07387	.200	.11182	.250	.15356

TABLE 7—CALCULATION OF WANTAGE IN CYLINDRICAL TANKS—Continued

<i>I+D</i>	<i>F</i>	<i>I+D</i>	<i>F</i>	<i>I+D</i>	<i>F</i>	<i>I+D</i>	<i>F</i>	<i>I+D</i>	<i>F</i>
.251	.15441	.301	.19908	.351	.24593	.401	.29435	.451	.34378
.252	.15528	.302	.20000	.352	.24689	.402	.29533	.452	.34477
.253	.15615	.303	.20092	.353	.24784	.403	.29631	.453	.34577
.254	.15702	.304	.20184	.354	.24880	.404	.29729	.454	.34676
.255	.15789	.305	.20276	.355	.24976	.405	.29827	.455	.34776
.256	.15876	.306	.20368	.356	.25071	.406	.29925	.456	.34875
.257	.15964	.307	.20460	.357	.25167	.407	.30024	.457	.34975
.258	.16051	.308	.20553	.358	.25263	.408	.30122	.458	.35075
.259	.16139	.309	.20645	.359	.25359	.409	.30220	.459	.35174
.260	.16226	.310	.20738	.360	.25455	.410	.30319	.460	.35274
.261	.16314	.311	.20830	.361	.25551	.411	.30417	.461	.35374
.262	.16402	.312	.20923	.362	.25647	.412	.30515	.462	.35474
.263	.16490	.313	.21015	.363	.25743	.413	.30614	.463	.35573
.264	.16578	.314	.21108	.364	.25839	.414	.30712	.464	.35673
.265	.16666	.315	.21201	.365	.25936	.415	.30811	.465	.35773
.266	.16755	.316	.21294	.366	.26032	.416	.30909	.466	.35872
.267	.16844	.317	.21387	.367	.26128	.417	.31108	.467	.35972
.268	.16931	.318	.21480	.368	.26225	.418	.31107	.468	.36072
.269	.17020	.319	.21573	.369	.26321	.419	.31205	.469	.36172
.270	.17109	.320	.21667	.370	.26418	.420	.31304	.470	.36272
.271	.17197	.321	.21760	.371	.26514	.421	.31403	.471	.36371
.272	.17287	.322	.21853	.372	.26611	.422	.31502	.472	.36471
.273	.17376	.323	.21947	.373	.26708	.423	.31600	.473	.36571
.274	.17465	.324	.22040	.374	.26804	.424	.31699	.474	.36671
.275	.17554	.325	.22134	.375	.26901	.425	.31798	.475	.36771
.276	.17643	.326	.22228	.376	.26998	.426	.31897	.476	.36871
.277	.17733	.327	.22321	.377	.27095	.427	.31996	.477	.36971
.278	.17822	.328	.22415	.378	.27192	.428	.32095	.478	.37071
.279	.17912	.329	.22509	.379	.27289	.429	.32194	.479	.3717
.280	.18002	.330	.22603	.380	.27386	.430	.32293	.480	.3727
.281	.18092	.331	.22697	.381	.27483	.431	.32391	.481	.3737
.282	.18182	.332	.22791	.382	.27580	.432	.32490	.482	.3747
.283	.18272	.333	.22886	.383	.27677	.433	.32590	.483	.3757
.284	.18361	.334	.22980	.384	.27775	.434	.32689	.484	.3767
.285	.18452	.335	.23074	.385	.27872	.435	.32788	.485	.3777
.286	.18542	.336	.23169	.386	.27969	.436	.32887	.486	.3787
.287	.18633	.337	.23263	.387	.28067	.437	.32987	.487	.3797
.288	.18723	.338	.23358	.388	.28164	.438	.33086	.488	.3807
.289	.18814	.339	.23453	.389	.28262	.439	.33185	.489	.3817
.290	.18905	.340	.23547	.390	.28359	.440	.33284	.490	.3827
.291	.18995	.341	.23642	.391	.28457	.441	.33384	.491	.3837
.292	.19086	.342	.23737	.392	.28554	.442	.33483	.492	.3847
.293	.19177	.343	.23832	.393	.28652	.443	.33582	.493	.3857
.294	.19268	.344	.23927	.394	.28750	.444	.33682	.494	.3867
.295	.19360	.345	.24022	.395	.28848	.445	.33781	.495	.3877
.296	.19451	.346	.24117	.396	.28945	.446	.33880	.496	.3887
.297	.19542	.347	.24212	.397	.29043	.447	.33980	.497	.3897
.298	.19634	.348	.24307	.398	.29141	.448	.34079	.498	.3907
.299	.19725	.349	.24403	.399	.29239	.449	.34179	.499	.3917
.300	.19817	.350	.24498	.400	.29337	.450	.34278	.500	.3927

TABLE 8

DENSITY (IN GRAMS PER MILLILITER) OF WATER AT TEMPERATURES  
FROM 0° TO 102° C \*

Temperature, Degrees C.	Density	Temperature, Degrees C.	Density	Temperature, Degrees C.	Density
0	0.99987	35	0.99406	70	0.97781
1	.99993	36	.99371	71	.97723
2	.99997	37	.99336	72	.97666
3	.99999	38	.99299	73	.97607
4	1.00000	39	.99262	74	.97548
5	.99999	40	.99224	75	.97489
6	.99997	41	.99186	76	.97428
7	.99993	42	.99147	77	.97368
8	.99988	43	.99107	78	.97307
9	.99981	44	.99066	79	.97245
10	.99973	45	.99024	80	.97183
11	.99963	46	.98982	81	.97120
12	.99952	47	.98940	82	.97057
13	.99940	48	.98896	83	.96994
14	.99927	49	.98852	84	.96930
15	.99913	50	.98807	85	.96865
16	.99897	51	.98762	86	.96800
17	.99880	52	.98715	87	.96734
18	.99862	53	.98669	88	.96668
19	.99843	54	.98621	89	.96601
20	.99823	55	.98573	90	.96534
21	.99802	56	.98524	91	.96467
22	.99780	57	.98478	92	.96399
23	.99756	58	.98425	93	.96330
24	.99732	59	.98375	94	.96261
25	.99707	60	.98324	95	.96192
26	.99681	61	.98272	96	.96122
27	.99654	62	.98220	97	.96051
28	.99626	63	.98167	98	.95981
29	.99597	64	.98113	99	.95909
30	.99567	65	.98059	100	.95838
31	.99537	66	.98005	101	.95765
32	.99505	67	.97950	102	.95693
33	.99473	68	.97894		
34	.99440	69	.97838		
35	.99406	70	.97781		

\*According to M. Thiesen, Wiss. Abh. der Physikalisch-Technischen Reichsanstalt,  
4, No. 1, 1904.

From Circular No. 19, U. S. Bureau of Standards.

TABLE 9

WEIGHT OF 1 CUBIC FOOT AND 1 GALLON (U. S. A. 231 CU. IN.) OF WATER <sup>1</sup>

Based on the water densities of P. Chappuis (Bureau International des Poids et Mesures, Travaux et Mémoires, XIII; 1907) for 0° to 40° C. and of M. Thiesen (Wis. Abh. der Physikalisch-Technischen Reichsanstalt, 4, No. 1; 1904) for 41° to 100° C. The weights in air are for dry air at the same temperature as the water up to 40° C. and at a (corrected) barometric pressure of 760 mm. and against brass weights of 8.4 density at 0° C. Above 40° C. the temperature of the air is assumed to be 20° C. The volumetric computations are based on the relation that 1 liter = 1.000027 cubic decimeters, and 1 cubic decimeter = 61.023378 cubic inches.

Temperature ° C.	Weight in Air			
	1 Cubic Foot		1 Gallon	
	Grams	Pounds	Grams	Pounds
0	28280.254	62.3472	3780.520	8.33461
1	82.033	.3511	0.758	.33613
2	83.321	.3540	0.930	.33551
3	84.121	.3557	1.037	.33575
4	84.458	.3565	1.082	.33585
5	84.345	.3562	1.067	.33582
6	83.784	.3550	0.992	.33565
7	82.804	.3528	0.861	.33536
8	81.413	.3498	0.675	.33495
9	79.618	.3458	0.435	.33442
10	77.441	.3410	0.144	.33378
11	74.853	.3353	3779.798	.33302
12	71.898	.3288	9.403	.33215
13	68.584	.3215	8.960	.33117
14	64.933	.3134	8.472	.33009
15 *	60.946	.3046	7.939	.32892
16 †	56.630	.2951	7.362	.32765
17	51.985	.2849	6.741	.32628
18	47.040	.2740	6.080	.32482
19	41.766	.2624	5.375	.32327
20	36.193	.2501	4.630	.32162
21	30.321	.2371	3.845	.31989
22	24.157	.2235	3.021	.31808
23	17.694	.2093	2.157	.31617
24	10.954	.1944	1.256	.31419

\* 15½ (60° F.)      28258.580      62.2994      3777.623      8.32820

† 16½ (62° F.)      28253.57      62.2884      3776.953      8.32670

<sup>1</sup> Calculated by the U. S. Bureau of Standards, Washington, January, 1915.

TABLE 9—WEIGHT OF 1 CUBIC FOOT AND 1 GALLON—Continued

Temperature ° C.	Weight in Air			
	1 Cubic Foot		1 Gallon	
	Grams	Pounds	Grams	Pounds
25	28203.930	62.1789	3770.317	8.31212
26	28196.629	.1629	3769.341	.30996
27	89.059	.1462	8.329	.30773
28	81.234	.1289	7.283	.30543
29	73.140	.1111	6.201	.30304
30	64.799	.0927	5.086	.30058
31	56.212	.0737	3.938	.29805
32	47.377	.0543	2.757	.29545
33	38.311	.0343	1.545	.29278
34	29.005	.0138	0.301	.29003
35	19.475	61.9926	3759.027	.28723
36	09.735	.9713	7.725	.28436
37	28099.771	.9493	6.393	.28142
38	89.596	.9269	5.033	.27842
39	79.215	.9040	3.645	.27536
40	68.645	.8807	2.232	.27225
41	55.68	.8520	0.50	.2684
42	44.62	.828	3749.02	.2652
43	33.30	.803	7.51	.2618
44	21.69	.777	5.96	.2584
45	09.80	.751	4.36	.2549
46	27997.90	.725	2.78	.2514
47	86.01	.698	1.18	.2479
48	73.54	.671	3739.52	.2442
49	61.09	.644	7.85	.2405
50	48.34	.615	6.15	.2368
51	35.60	.587	4.45	.2330
52	22.29	.558	2.67	.2291
53	09.26	.529	0.93	.2253
54	27895.66	.499	3729.11	.2213
55	82.07	.469	7.29	.2173
56	68.20	.439	5.44	.2132
57	55.17	.410	3.70	.2093
58	40.15	.377	1.69	.2049
59	26.00	.346	3719.80	.2007
60	11.55	.314	7.86	.1965
61	27796.83	.281	5.90	.1921
62	82.10	.249	3.93	.1878
63	67.09	.216	1.92	.1834
64	51.80	.182	3709.88	.1789

TABLE 9—*Continued*TABLE 9—WEIGHT OF 1 CUBIC FOOT AND 1 GALLON—*Continued*

Temperature ° C.	Weight in Air			
	1 Cubic Foot		1 Gallon	
	Grams	Pounds	Grams	Pounds
65	27736.50	61.148	3707.83	8.1744
66	21.21	.115	5.79	.1699
67	05.64	.080	3.70	.1653
68	27689.79	.046	1.58	.1806
69	73.91	.010	3699.46	.1559
70	57.78	60.975	7.31	.1512
71	41.35	.939	5.11	.1463
72	25.21	.903	2.95	.1418
73	08.50	.866	0.72	.1366
74	27591.79	.829	3688.49	.1317
75	75.08	.793	3686.25	.1268
76	57.81	.754	3.94	.1217
77	40.81	.717	1.67	.1167
78	23.53	.679	3679.36	.1116
79	05.97	.640	7.01	.1064
80	27488.42	.602	4.67	.1012
81	70.58	.562	2.28	.0960
82	52.74	.523	3669.90	.0907
83	34.90	.484	7.51	.0855
84	16.77	.444	5.09	.0801
85	27398.36	.403	2.63	.0747
86	79.96	.362	0.17	.0693
87	61.26	.321	3657.67	.0638
88	42.57	.280	5.17	.0583
89	23.59	.238	2.63	.0527
90	04.62	.196	0.10	.0471
91	27285.65	.154	3647.56	.0415
92	66.38	.112	4.99	.0358
93	46.85	.069	2.37	.0301
94	27.31	.026	3639.76	.0243
95	07.76	59.983	7.15	.0185
96	27187.94	.939	4.50	.0127
97	67.84	.895	1.81	.0068
98	48.01	.851	3629.16	.0009
99	27.62	.806	6.44	7.9949
100	07.51	.762	3.75	.9889



TABLE 10

FOR THE CALIBRATION OF FLASKS TO TRUE CUBIC CENTIMETERS AT 20° C.\*

Apparent Weight (in Grains) of Water in Air

[This table gives the apparent weight for temperature between 15° and 30° C., humidity 50 per cent, unreduced barometer reading 76 cm., of certain volumes of water weighed with brass weights. The table may be conveniently employed to determine definite volumes of water for calibrating instruments. The air is assumed to be at the same temperature as the water.]

Temp. ° C.	2000 ml.	1000 ml.	500 ml.	400 ml.	300 ml.	250 ml.	150 ml.
15	1996.11	998.05	499.03	399.22	299.42	249.51	149.71
16	1995.80	997.90	498.95	399.16	299.37	249.43	149.68
17	1995.48	997.74	498.87	399.10	299.32	249.43	149.66
18	1995.13	997.56	498.78	399.03	299.27	249.39	149.63
19	1994.76	997.38	498.69	398.95	299.21	249.34	149.61
20	1994.36	997.18	498.59	398.87	299.15	249.30	149.58
21	1993.95	996.97	498.49	398.79	299.09	249.24	149.55
22	1993.51	996.76	498.38	398.70	299.03	249.19	149.51
23	1993.06	996.53	498.26	398.61	298.96	249.13	149.48
24	1992.58	996.29	498.15	398.52	298.89	249.07	149.44
25	1992.09	996.04	498.02	398.42	298.81	249.01	149.41
26	1991.57	995.79	497.89	398.31	298.74	248.95	149.37
27	1991.04	995.52	497.76	398.21	298.66	248.88	149.33
28	1990.49	995.24	497.62	398.10	298.57	248.81	149.29
29	1989.92	994.96	497.48	397.98	298.49	248.74	149.24
30	1989.33	994.66	497.33	397.87	298.40	248.67	149.20

\* From Circular No. 19, U. S. Bureau of Standards, April 1, 1914.

TABLE 10A

SHOWING THE VOLUME OF SUGAR SOLUTIONS AT DIFFERENT TEMPERATURES.—(GERLACH.)

Temp. ° C.	10 per cent	20 per cent	30 per cent	40 per cent	50 per cent
0	10000	10000	10000	10000	10000
5	10004.5	10007	10009	10012	10016
10	10012	10016	10021	10026	10032
15	10021	10028	10034	10042	10050
20	10033	10041	10049	10058	10069
25	10048	10057	10066	10075	10088
30	10064	10074	10084	10094	10110
35	10082	10092	10103	10114	10132
40	10101	10112	10124	10136	10156
45	10122	10134	10146	10160	10180
50	10145	10158	10170	10184	10204
55	10170	10183	10196	10210	10229
60	10197	10209	10222	10235	10253
65	10225	10236	10249	10261	10278
70	10255	10265	10277	10287	10306
75	10284	10295	10306	10316	10332
80	10316	10325	10335	10345	10360
85	10347	10355	10365	10375	10388
90	10379	10387	10395	10405	10417
95	10411	10418	10425	10435	10445
100	10442	10450	10458	10465	10457

TABLE 11

TABLES OF CORRECTIONS FOR DETERMINING THE TRUE CAPACITIES OF FLASKS  
FROM THE WEIGHT OF WATER IN AIR \*

(Following data are assumed as approximating ordinary conditions: Observed barometric pressure = 76 cm.; relative humidity = 50 per cent; coefficient of expansion of glass = 0.000025 per degree C.)

## INDICATED CAPACITY 50 ML.

Temp. ° C.	Tenths of Degrees									
	0	1	2	3	4	5	6	7	8	9
15	0.104	0.104	0.105	0.106	0.106	0.107	0.107	0.108	0.109	0.109
16	.110	.111	.111	.112	.113	.113	.114	.115	.116	.116
17	.117	.118	.118	.119	.120	.121	.121	.122	.123	.124
18	.124	.125	.126	.127	.128	.128	.129	.130	.131	.132
19	.132	.133	.134	.135	.136	.137	.137	.138	.139	.140
20	.141	.142	.143	.144	.144	.145	.146	.147	.148	.149
21	.150	.151	.152	.153	.154	.155	.156	.157	.158	.159
22	.160	.161	.162	.163	.164	.165	.166	.167	.168	.169
23	.170	.171	.172	.173	.174	.175	.176	.177	.178	.179
24	.180	.182	.183	.184	.185	.186	.187	.188	.189	.190
25	.192	.193	.194	.195	.196	.197	.199	.200	.201	.202
26	.203	.204	.206	.207	.208	.209	.210	.212	.213	.214
27	.215	.216	.218	.219	.220	.222	.223	.224	.225	.226
28	.228	.229	.230	.232	.233	.234	.236	.237	.238	.240
29	.241	.242	.244							

## INDICATED CAPACITY 100 ML.

Temp. ° C.	Tenths of Degrees									
	0	1	2	3	4	5	6	7	8	9
15	0.207	0.208	0.210	0.211	0.212	0.213	0.215	0.216	0.217	0.219
16	.220	.221	.223	.224	.225	.227	.228	.230	.231	.232
17	.234	.235	.237	.238	.240	.241	.243	.244	.246	.247
18	.249	.250	.252	.253	.255	.257	.258	.260	.261	.263
19	.265	.266	.268	.270	.272	.273	.275	.277	.278	.280
20	.282	.284	.285	.287	.289	.291	.293	.294	.296	.298
21	.300	.302	.304	.306	.308	.310	.311	.314	.315	.317
22	.319	.321	.323	.325	.327	.329	.331	.333	.336	.338
23	.340	.342	.344	.346	.348	.350	.352	.354	.357	.359
24	.361	.363	.365	.368	.370	.372	.374	.376	.379	.381
25	.383	.386	.388	.390	.392	.395	.397	.399	.402	.404
26	.406	.409	.411	.414	.416	.418	.421	.423	.426	.428
27	.431	.433	.436	.438	.440	.443	.446	.448	.451	.453
28	.456	.458	.461	.463	.466	.469	.471	.474	.476	.479
29	.482	.484	.487							

The tables of corrections give for each nominal capacity and observed temperature the amounts to be added to the apparent weight (in air against brass weights) of the water contained by the flask to give the capacity in True Cubic Centimeters at 20° C. Example: Apparent weight of water at 22.3° = 99.68; adding correction 0.325 = 100.005, the actual capacity.

\* From Circular No. 19, U. S. Bureau of Standards, April 1, 1914.

TABLE 12

FOR THE CALIBRATION OF FLASKS TO MOHR'S CUBIC CENTIMETERS

(This table has been calculated from the data given in U. S. Bureau of Standards Circular No. 19, April, 1914.)

Temp. ° C.	Apparent Weight, Grams	Temp. ° C.	Apparent Weight, Grams	Temp. ° C.	Apparent Weight, Grams	Temp. ° C.	Apparent Weight, Grams
15	100.033	19	99.975	24	99.879	29	99.758
16	100.020	20	99.958	25	99.857	30	99.731
17	100.007	21	99.940	26	99.834	31	99.704
17.5	100.000	22	99.921	27	99.809	32	99.674
18	99.991	23	99.900	28	99.784	33	99.644

Mohr's cubic centimeter or unit is the volume occupied by 1 gram of water as weighed with brass weights in the air at 17.5° C. Flasks graduated to Mohr's cubic centimeter should be used with the normal weight of 26.048 grams. This table is designed to obviate the necessity of making the check calibrations at 17.5° C. (See page 217.)

TABLE 13

SHOWING THE BOILING-POINTS OF SUGAR SOLUTIONS—(FLOURENO, FREUTZEL)  
SUCROSE SOLUTIONS

Per Cent Sucrose	Boiling-point, ° C.	Per Cent Sucrose	Boiling-point, ° C.	Per Cent Sucrose	Boiling-point, ° C.
10	100.1	55	102.4	75	107.4
20	100.3	60	103.1	80	110.3
30	100.6	65	103.9	85	114.5
40	101.1	70	105.3	90	122.6
50	101.9				

## JUICE, SIRUP AND MOLASSES

Degree Brix	Boiling-point, ° C.		Degree Brix	Boiling-point, ° C.	
	Juice and Sirup	Molasses		Juice and Sirup	Molasses
10	100.2	100.3	55	102.8	103.4
20	100.2	100.6	60	103.5	104.2
30	100.8	101.1	65	104.4	105.3
40	101.4	101.7	70	105.8	106.8
50	102.2	102.7	75		

TABLE 14

SPECIFIC GRAVITIES OF COMMON ACIDS AND ALKALIES. AQUEOUS SOLUTIONS  
(From Int. Critical Tables, 1928)

Direct interpolation may be used for fractions of per cents

Per Cent Material by Weight	HCl	HNO <sub>3</sub>	H <sub>2</sub> SO <sub>4</sub>	H <sub>3</sub> PO <sub>4</sub>	NaOH	KOH	NH <sub>3</sub>
	20°/4°	20°/4°	20°/4°	20°/4°	20°/4°	15°/4°	20°/4°
1	1.0032	1.00364	1.0051	1.0038	1.0095	1.0083	0.9989
2	1.0082	1.00909	1.0118	1.0092	1.0207	1.0175	0.9895
3	.....	1.01457	1.0184	.....	1.0318	1.0267	.....
4	1.0181	1.02008	1.0250	1.0200	1.0428	1.0359	0.9811
5	.....	1.02563	1.0317	.....	1.0538	1.0452	.....
6	1.0279	1.03122	1.0385	1.0309	1.0648	1.0544	0.9730
7	.....	1.0369	1.0453	.....	1.0758	1.0637	.....
8	1.0376	1.0427	1.0522	1.0420	1.0869	1.0730	0.9651
9	.....	1.0485	1.0591	.....	1.0979	1.0824	.....
10	1.0474	1.0543	1.0661	1.0532	1.1089	1.0918	0.9575
11	.....	1.0602	1.0731	.....	.....	1.1013	.....
12	1.0574	1.0661	1.0802	1.0647	1.1309	1.1108	0.9501
13	.....	1.0721	1.0874	.....	.....	1.1203	.....
14	1.0675	1.0781	1.0947	1.0764	1.1530	1.1299	0.9430
15	.....	1.0842	1.1020	.....	.....	1.1396	.....
16	1.0776	1.0903	1.1094	1.0884	1.1751	1.1493	0.9362
17	.....	1.0964	1.1168	.....	.....	1.1590	.....
18	1.0878	1.1026	1.1243	1.1008	1.1972	1.1688	0.9295
19	.....	1.1088	1.1318	.....	.....	1.1786	.....
20	1.0980	1.1150	1.1394	1.1134	1.2191	1.1884	0.9229
21	.....	1.1213	1.1471	.....	.....	1.1984	.....
22	1.1083	1.1276	1.1548	1.1263	1.2411	1.2083	0.9164
23	.....	1.1340	1.1626	.....	.....	1.2184	.....
24	1.1187	1.1404	1.1704	1.1395	1.2629	1.2285	0.9101
25	.....	1.1469	1.1783	.....	.....	1.2387	.....
26	1.1290	1.1534	1.1862	1.1529	1.2848	1.2489	0.9040
27	.....	1.1600	1.1942	.....	.....	1.2592	.....
28	1.1392	1.1666	1.2023	1.1665	1.3064	1.2695	0.8980
29	.....	1.1733	1.2104	.....	.....	1.2800	.....
30	1.1493	1.1800	1.2185	1.1805	1.3279	1.2905	0.8920
31	.....	1.1867	1.2267	.....	.....	1.3010	.....
32	1.1593	1.1934	1.2349	.....	1.3490	1.3117	.....
33	.....	1.2002	1.2432	.....	.....	1.3224	.....
34	1.1691	1.2071	1.2515	.....	1.3696	1.3331	.....
35	.....	1.2140	1.2599	1.216	.....	1.3440	.....
36	1.1789	1.2205	1.2684	.....	1.3900	1.3549	.....
37	.....	1.2270	1.2769	.....	.....	1.3659	.....
38	1.1885	1.2325	1.2855	.....	1.4101	1.3769	.....
39	.....	1.2399	1.2941	.....	.....	1.3879	.....
40	1.1980	1.2463	1.3028	1.254	1.4300	1.3991	.....
45	.....	1.2783	1.3476	1.293	.....	1.4558	.....
50	.....	1.3100	1.3951	1.355	1.5253	.....	.....
55	.....	1.3393	1.4453	1.379	.....	.....	.....
60	.....	1.3667	1.4983	1.426	.....	.....	.....
70	.....	1.4134	1.6105	.....	.....	.....	.....
80	.....	1.4521	1.7272	.....	.....	.....	.....
90	.....	1.4826	1.8144	.....	.....	.....	.....
100	.....	1.5217	1.8305	.....	.....	.....	.....

TABLE 15

SHOWING THE AMOUNT OF CaO IN MILK OF LIME OF VARIOUS DENSITIES AT 15° C.  
(From Blatner's Table)

Degree Brix	Degree Baumé	Weight of one Liter, Milk of Lime, Grams	CaO per Liter, Grams	Per Cent CaO	Degree Brix	Degree Baumé	Weight of One Liter, Milk of Lime, Grams	CaO per Liter, Grams	Per Cent CaO
1.8	1	1007	7.5	0.745	29	16	1125	159	14.13
3.6	2	1014	16.5	1.64	30.8	17	1134	170	15
5.4	3	1022	26	2.54	32.7	18	1142	181	15.85
7.2	4	1029	36	3.5	34.6	19	1152	193	16.75
9	5	1037	46	4.43	36.4	20	1162	206	17.72
10.8	6	1045	56	5.36	38.3	21	1171	218	18.61
12.6	7	1052	65	6.18	40.1	22	1180	229	19.4
14.4	8	1060	75	7.08	42	23	1190	242	20.34
16.2	9	1067	84	7.87	43.9	24	1200	255	21.25
18	10	1075	94	8.74	45.8	25	1210	268	22.15
19.8	11	1083	104	9.6	47.7	26	1220	281	23.03
21.7	12	1091	115	10.54	49.6	27	1231	295	23.96
23.5	13	1100	126	11.45	51.5	28	1241	309	24.9
25.3	14	1108	137	12.35	53.5	29	1252	324	25.87
27.2	15	1116	148	13.26	55.4	30	1263	339	26.84

TABLE 16

SHOWING LITERS OF MILK OF LIME OF VARIOUS DENSITIES FOR 1 KILO OF CaO  
—(MATEGECZEK)

Degree Brix	Degree Baumé	1 Kilo CaO per .. Liters Milk of Lime	Degree Brix	Degree Baumé	1 Kilo CaO per .. Liters Milk of Lime
18	10	7.50	38.3	21	4.28
20	11	7.10	40.2	22	4.16
21.7	12	6.70	42.0	23	4.05
23.5	13	6.30	43.9	24	3.95
25.3	14	5.88	45.8	25	3.87
27.2	15	5.50	47.7	26	3.81
29	16	5.25	49.6	27	3.75
30.9	17	5.01	51.6	28	3.70
32.7	18	4.80	53.5	29	3.65
34.6	19	4.68	55.5	30	3.60
36.5	20	4.42			

TABLE 17

SHOWING THE PERCENTAGE OF LEAD ACETATE IN SOLUTIONS OF THE SALT, OF DIFFERENT DENSITIES, AT 15° C.—(GERLACH)

Specific Gravity	Per Cent of the Salt	Specific Gravity	Per Cent of the Salt	Specific Gravity	Per Cent of the Salt
1.0127	2	1.1384	20	1.2768	36
1.0255	4	1.1544	22	1.2966	38
1.0386	6	1.1704	24	1.3163	40
1.0520	8	1.1869	26	1.3376	42
1.0654	10	1.2040	28	1.3588	44
1.0796	12	1.2211	30	1.3810	46
1.0939	14	1.2395	32	1.4041	48
1.1084	16	1.2578	34	1.4271	50
1.1234	18				

TABLE 18

SHOWING THE SOLUBILITY OF BARIUM OXIDE IN SUGAR SOLUTIONS  
(Pellet and Sencier, *La fabrication du sucre*, 1, 186)

Sucrose per 100 Cc.	Baryta (BaO) per 100 Cc.	Baryta (BaO) Per Cent Sucrose
2.5	4.59	18.3
5	5.46	10.9
7.5	6.56	8.7
10	7.96	7.7
12.5	9.41	7.5
15	10.00	6.6
20	10.90	5.4
25	12.90	5.1
30	14.68	4.9

TABLE 19

SHOWING THE SOLUBILITY OF CERTAIN SALTS IN WATER IN THE PRESENCE OF SUCROSE  
(Jacobsthal, *Zeit. Rübenzuckerind.*, 18, 649; taken from Sidersky's *Traité d'analyse des Matières Sucrées*, p. 11)

Solution containing	5% Sucrose, Grams	10% Sucrose, Grams	15% Sucrose, Grams	20% Sucrose, Grams	25% Sucrose, Grams
Calcium sulphate.....	2.095	1.946	1.593	1.539	1.333
Calcium carbonate.....	0.027	0.036	0.024	0.022	0.008
Calcium oxalate.....	0.033	0.047	0.012	0.008	0.001
Calcium phosphate.....	0.029	0.028	0.014	0.018	0.005
Calcium citrate.....	1.813	1.578	1.505	1.454	1.454
Magnesium carbonate...	0.317	0.199	0.194	0.213	0.284

TABLE 20

SHOWING THE SOLUBILITY OF SUGAR IN ALCOHOL AT 17.5° C.—(OTTO SCHREFELD)

(Zell. f. Rübenzucker-Ind., 44, 970)

Alcohol Per Cent by Weight	Sucrose Per Cent	Sucrose in Grams in 100 Grams of the Mixture of Alcohol and Water Solution
0	66.20	195.8
5*	64.25	179.7
10*	62.20	164.5
15	60.40	152.5
20*	58.55	141.2
25	56.20	128.3
30	54.05	117.8
35	51.25	105.3
40	47.75	91.3
45	43.40	76.6
50	38.55	62.7
55	32.80	48.8
60	26.70	36.4
65	19.50	24.2
70	12.25	13.9
75	7.20	7.7
80	4.05	4.2
85	2.10	2.1
90	0.95	0.09
95	0.15	0.01
Absolute	0.00	0.00

\* Calculated.

TABLE 21

SHOWING THE SOLUBILITY OF STRONTIUM OXIDE IN SUGAR SOLUTIONS.—(SIDERSKY)

Per Cent Sucrose	Strontia (SrO) Per Cent of the Solution				Per Cent Sucrose	Strontia (SrO) Per Cent of the Solution			
	At 3° C.	At 15° C.	At 24° C.	At 40° C.		At 3° C.	At 15° C.	At 24° C.	At 40° C.
1	0.45	0.65	0.70	1.68	11	1.30	1.57	2.01	3.75
2	0.53	0.75	0.83	1.89	12	1.38	1.66	2.14	3.96
3	0.62	0.84	0.96	2.09	13	1.47	1.75	2.28	4.16
4	0.70	0.93	1.09	2.30	14	1.55	1.84	2.41	4.37
5	0.79	1.03	1.22	2.51	15	1.64	1.94	2.55	4.58
6	0.87	1.12	1.35	2.72	16	1.72	2.03	2.69	4.79
7	0.96	1.21	1.48	2.92	17	1.81	2.12	2.83	4.99
8	1.04	1.30	1.61	3.13	18	1.90	2.21	2.97	5.20
9	1.13	1.39	1.74	3.33	19	1.99	2.30	3.11	5.41
10	1.21	1.48	1.87	3.55	20	2.08	2.39	3.25	5.61

TABLE 22

SHOWING THE SOLUBILITY OF SUGAR IN WATER.—(HERZFELD)

Temp., ° C.	Sugar, Per Cent.	Temp., ° C.	Sugar, Per Cent.	Temp., ° C.	Sugar, Per Cent.
0	64.18	35	69.55	70	76.22
5	64.87	40	70.42	75	77.27
10	65.58	45	71.32	80	78.36
15	66.53	50	72.25	85	79.46
20	67.09	55	73.20	90	80.61
25	67.89	60	74.18	95	81.77
30	68.80	65	75.88	100	82.97

The solubility is decreased by presence of a small quantity of organic or inorganic salts, but increased by a large quantity.

TABLE 23

SHOWING THE SOLUBILITY OF LIME IN SOLUTIONS OF SUGAR

Sugar in 100 Parts Water	Density of Sirup	Density after Saturation with Lime	100 Parts of the Residue Dried at 120° C. Contain:	
			Lime	Sugar
40	1.122	1.179	21	79
35	1.110	1.166	20.5	79.5
30	1.096	1.148	20.1	79.9
25	1.082	1.128	19.8	80.2
20	1.068	1.104	18.8	81.2
15	1.052	1.080	18.5	81.5
10	1.036	1.053	18.1	81.9
5	1.018	1.026	15.3	84.7

TABLE 24

SHOWING THE CONTRACTION OF INVERT SUGAR ON DISSOLVING IN WATER; ALSO, THE CONTRACTION OF CANE-SUGAR SOLUTIONS ON INVERSION

(From "Manuel Agenda," Gallois and Dupont)

Per Cent Sugar	Volume	Contraction	Specific Gravity	
			Cane-sugar Solution	Invert-sugar Solution
0	1.00000	0.00000	1.0000	1.0000
5	.99863	0.00137	1.0203	1.0206
10	.99744	0.00256	1.0413	1.0418
15	.99639	0.00361	1.0630	1.0631
20	.99546	0.00454	1.0854	1.0856
25	.99462	0.00538	1.1086	1.1086



TABLE 25

SHOWING THE EVAPORATION OF WATER IN CONCENTRATING JUICE TO SIRUP.—  
(SPENCER)

(Percentages by Weight). (See 391)

Brix of Juice	Degree Brix of the Sirup and the Evaporation in Percentage Terms of the Juice									
	52.0	52.5	53.0	53.5	54.0	54.5	55.0	55.5	56.0	56.5
11.0	78.8	79.0	79.2	79.4	79.6	79.8	80.0	80.2	80.4	80.5
.2	78.4	78.6	78.8	79.0	79.2	79.4	79.6	79.8	80.0	80.2
.4	78.0	78.2	78.5	78.7	78.9	79.0	79.3	79.5	79.7	79.8
.6	77.7	77.9	78.1	78.3	78.5	78.7	78.9	79.1	79.3	79.5
.8	77.3	77.5	77.7	78.0	78.2	78.4	78.6	78.5	79.0	79.1
12.0	76.9	77.1	77.4	77.6	77.8	78.0	78.2	78.4	78.6	78.8
.2	76.5	76.7	77.0	77.2	77.4	77.6	77.8	78.0	78.2	78.4
.4	76.1	76.3	76.6	76.8	77.0	77.2	77.5	77.7	77.9	78.1
.6	75.8	76.0	76.3	76.5	76.7	76.9	77.2	77.4	77.6	77.8
.8	75.4	75.6	75.9	76.1	76.3	76.5	76.8	77.0	77.2	77.4
13.0	75.0	75.2	75.5	75.7	75.9	76.1	76.4	76.6	76.8	77.0
.2	74.6	74.8	75.1	75.3	75.5	75.7	76.0	76.2	76.4	76.6
.4	74.2	74.4	74.7	74.9	75.1	75.3	75.6	75.8	76.0	76.2
.6	73.9	74.1	74.4	74.6	74.8	75.0	75.2	75.4	75.6	75.8
.8	73.5	73.7	74.0	74.2	74.4	74.6	74.8	75.0	75.2	75.4
14.0	73.1	73.3	73.6	73.8	74.1	74.3	74.6	74.8	75.0	75.2
.2	72.7	72.9	73.2	73.4	73.7	73.9	74.2	74.4	74.6	74.8
.4	72.3	72.5	72.9	73.1	73.3	73.6	73.8	74.0	74.2	74.4
.6	72.0	72.1	72.5	72.7	72.9	73.2	73.5	73.7	74.0	74.2
.8	71.6	71.8	72.1	72.3	72.6	72.8	73.2	73.4	73.6	73.8
15.0	71.2	71.4	71.7	71.9	72.2	72.5	72.8	73.0	73.2	73.4
.2	70.8	71.0	71.3	71.5	71.8	72.1	72.4	72.6	72.8	73.0
.4	70.4	70.6	70.9	71.1	71.4	71.8	72.1	72.3	72.5	72.7
.6	70.0	70.3	70.6	70.8	71.0	71.4	71.7	71.9	72.1	72.3
.8	69.6	69.9	70.2	70.4	70.7	71.0	71.3	71.5	71.7	71.9
16.0	69.2	69.5	69.8	70.1	70.4	70.7	70.9	71.2	71.4	71.7
.2	68.8	69.1	69.4	69.7	70.0	70.2	70.5	70.8	71.1	71.3
.4	68.4	68.7	69.1	69.4	69.6	69.9	70.2	70.4	70.7	70.9
.6	68.0	68.3	68.7	69.0	69.3	69.5	69.8	70.1	70.4	70.6
.8	67.7	68.0	68.3	68.6	68.9	69.2	69.4	69.7	70.0	70.3
17.0	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	69.9
.2	66.9	67.2	67.5	67.8	68.1	68.4	68.8	69.1	69.4	69.6
.4	66.5	66.8	67.1	67.4	67.7	68.0	68.4	68.7	69.1	69.3
.6	66.2	66.5	66.8	67.1	67.4	67.7	68.0	68.3	68.6	68.8
.8	65.8	66.1	66.4	66.7	67.0	67.3	67.7	68.0	68.3	68.5
18.0	65.4	65.7	66.0	66.4	66.7	67.0	67.3	67.6	67.9	68.2
.2	65.0	65.3	65.6	66.0	66.3	66.6	66.9	67.2	67.5	67.8
.4	64.6	64.9	65.2	65.6	65.9	66.2	66.6	66.9	67.2	67.5
.6	64.2	64.5	64.8	65.2	65.5	65.8	66.2	66.5	66.8	67.1
.8	63.8	64.1	64.4	64.8	65.1	65.4	65.8	66.1	66.4	66.7
19.0	63.4	63.8	64.2	64.5	64.8	65.1	65.4	65.8	66.1	66.4
.2	63.0	63.4	63.8	64.1	64.4	64.7	65.0	65.4	65.7	66.0
.4	62.6	63.0	63.4	63.7	64.0	64.3	64.7	65.1	65.4	65.7
.6	62.3	62.7	63.1	63.4	63.7	64.0	64.3	64.7	65.0	65.3
.8	61.9	62.3	62.7	63.0	63.3	63.6	64.0	64.4	64.7	65.0
20.0	61.5	61.9	62.3	62.6	63.0	63.3	63.6	64.0	64.3	64.6
.2	61.1	61.5	61.9	62.2	62.6	62.9	63.2	63.6	63.9	64.2
.4	60.7	61.1	61.5	61.8	62.2	62.5	62.9	63.3	63.6	63.9
.6	60.4	60.8	61.2	61.5	61.9	62.2	62.6	63.0	63.3	63.6
.8	60.0	60.4	60.8	61.1	61.5	61.8	62.2	62.6	62.9	63.2
21.0	59.6	60.0	60.4	60.8	61.1	61.5	61.8	62.2	62.5	62.8

TABLE 25—Continued

SHOWING THE EVAPORATION OF WATER IN CONCENTRATING JUICE TO SIRUP.—  
(SPENCER)

Brix of Juice	Degree Brix of the Sirup and the Evaporation in Percentage Terms of the Juice									
	57.0	57.5	58.0	58.5	59.0	59.5	60.0	60.5	61.0	61.5
11.0	80.7	80.9	81.0	81.2	81.4	81.5	81.7	81.8	82.0	82.1
.2	80.3	80.5	80.7	80.9	81.1	81.2	81.4	81.5	81.7	81.8
.4	80.0	80.2	80.4	80.5	80.7	80.8	81.0	81.2	81.3	81.5
.6	79.6	79.8	80.1	80.2	80.3	80.5	80.7	80.8	81.0	81.1
.8	79.3	79.5	79.7	79.9	80.0	80.2	80.3	80.5	80.7	80.8
12.0	79.0	79.2	79.3	79.5	79.7	79.9	80.0	80.2	80.4	80.5
.2	78.6	78.8	79.0	79.1	79.3	79.5	79.7	79.9	80.0	80.1
.4	78.3	78.5	78.6	78.8	78.9	79.2	79.3	79.5	79.7	79.8
.6	78.0	78.2	78.3	78.5	78.6	78.8	79.0	79.2	79.4	79.5
.8	77.6	77.8	77.9	78.1	78.3	78.5	78.7	78.8	79.0	79.2
13.0	77.2	77.4	77.6	77.8	78.0	78.2	78.3	78.5	78.7	78.9
.2	76.8	77.0	77.2	77.4	77.6	77.8	78.0	78.2	78.4	78.5
.4	76.4	76.6	76.9	77.1	77.3	77.5	77.7	77.9	78.0	78.2
.6	76.0	76.2	76.5	76.8	77.0	77.2	77.3	77.5	77.5	77.9
.8	75.7	75.8	76.2	76.4	76.6	76.8	77.0	77.2	77.4	77.5
14.0	75.4	75.6	75.9	76.1	76.3	76.5	76.7	76.9	77.0	77.2
.2	75.0	75.2	75.5	75.7	75.9	76.1	76.3	76.5	76.7	76.9
.4	74.6	74.8	75.2	75.4	75.6	75.8	76.0	76.2	76.4	76.6
.6	74.4	74.6	74.8	75.0	75.2	75.5	75.7	75.9	76.1	76.3
.8	74.0	74.2	74.5	74.7	74.9	75.1	75.3	75.7	75.7	75.9
15.0	73.7	73.9	74.2	74.4	74.6	74.8	75.0	75.2	75.4	75.6
.2	73.2	73.4	73.8	74.0	74.2	74.5	74.7	74.9	75.1	75.3
.4	72.9	73.1	73.5	73.7	73.9	74.1	74.3	74.5	74.8	74.9
.6	72.5	72.7	73.1	73.3	73.5	73.8	74.0	74.2	74.4	74.6
.8	72.1	72.3	72.8	73.0	73.2	73.5	73.7	73.9	74.1	74.3
16.0	71.9	72.1	72.4	72.6	72.9	73.1	73.3	73.6	73.8	74.0
.2	71.5	71.7	72.1	72.3	72.5	72.8	73.0	73.2	73.5	73.6
.4	71.1	71.3	71.7	72.0	72.2	72.4	72.7	72.9	73.1	73.3
.6	70.9	71.1	71.4	71.6	71.9	72.1	72.3	72.5	72.8	73.0
.8	70.5	70.8	71.0	71.3	71.5	71.8	72.0	72.2	72.5	72.7
17.0	70.2	70.4	70.7	71.0	71.2	71.5	71.7	71.9	72.1	72.4
.2	69.8	70.0	70.3	70.6	70.8	71.1	71.3	71.6	71.8	72.0
.4	69.5	69.7	70.0	70.3	70.5	70.8	71.0	71.2	71.5	71.7
.6	69.0	69.2	69.7	69.9	70.2	70.4	70.7	70.9	71.1	71.4
.8	68.7	68.9	69.3	69.6	69.8	70.1	70.3	70.6	70.8	71.1
18.0	68.4	68.7	69.0	69.2	69.5	69.8	70.0	70.3	70.5	70.7
.2	68.0	68.3	68.6	68.9	69.1	69.4	69.7	69.9	70.1	70.4
.4	67.7	68.0	68.3	68.5	68.8	69.1	69.3	69.6	69.8	70.1
.6	67.3	67.6	67.9	68.2	68.5	68.7	69.0	69.2	69.5	69.8
.8	67.0	67.3	67.6	67.9	68.1	68.4	68.7	68.9	69.1	69.4
19.0	66.7	67.0	67.2	67.5	67.8	68.1	68.3	68.6	68.8	69.1
.2	66.3	66.6	66.9	67.2	67.5	67.7	68.0	68.2	68.5	68.8
.4	66.0	66.3	66.5	66.8	67.1	67.4	67.7	67.9	68.2	68.5
.6	65.6	65.9	66.2	66.5	66.8	67.0	67.3	67.6	67.8	68.1
.8	65.3	65.6	65.9	66.2	66.4	66.7	67.0	67.3	67.5	67.8
20.0	64.9	65.2	65.5	65.8	66.1	66.4	66.7	67.0	67.2	67.5
.2	64.5	64.8	65.2	65.5	65.7	66.0	66.3	66.6	66.9	67.2
.4	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.8
.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	65.9	66.2	66.5
.8	63.5	63.8	64.1	64.4	64.7	65.1	65.3	65.6	65.9	66.2
21.0	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.8

TABLE 26

SHOWING THE EVAPORATION OF WATER IN CONCENTRATING JUICE TO SIRUP (SPENCER)  
(Percentages by Volume). (See 391)

Brix of Juice	Degree Brix of the Sirup and the Evaporation in Percentage Terms of the Juice																				
	53.0	53.5	54.0	54.5	55.0	55.5	56.0	56.5	57.0	57.5	58.0	58.5	59.0	59.5	60.0	60.5	61.0	61.5	62.0	62.5	63.0
10.0	84.3	84.5	84.7	84.9	85.0	85.2	85.3	85.5	85.7	85.9	86.0	86.2	86.3	86.5	86.6	86.7	86.8	86.9	87.1	87.3	87.4
.5	83.5	83.7	83.9	84.1	84.3	84.5	84.6	84.8	85.0	85.2	85.3	85.4	85.6	85.8	85.9	86.0	86.1	86.2	86.4	86.6	86.7
11.0	82.7	82.9	83.1	83.3	83.5	83.7	83.8	84.0	84.2	84.4	84.6	84.7	84.9	85.0	85.1	85.3	85.4	85.6	85.8	86.0	86.1
.5	81.9	82.1	82.3	82.5	82.7	82.9	83.0	83.3	83.5	83.7	83.9	84.0	84.1	84.3	84.4	84.6	84.7	84.9	85.1	85.3	85.4
12.0	81.0	81.2	81.4	81.6	81.9	82.1	82.2	82.5	82.7	82.9	83.1	83.3	83.4	83.6	83.7	83.9	84.1	84.3	84.4	84.6	84.7
.5	80.2	80.4	80.6	80.9	81.1	81.3	81.5	81.8	82.0	82.2	82.4	82.5	82.7	82.9	83.0	83.2	83.4	83.6	83.7	83.9	84.1
13.0	79.3	79.6	79.8	80.1	80.3	80.5	80.7	81.0	81.2	81.4	81.6	81.8	82.0	82.2	82.3	82.5	82.7	82.9	83.1	83.3	83.5
.5	78.5	78.8	79.0	79.3	79.5	79.7	79.9	80.2	80.4	80.6	80.8	81.1	81.3	81.5	81.6	81.8	82.0	82.2	82.4	82.6	82.8
14.0	77.7	77.9	78.1	78.4	78.7	78.9	79.1	79.4	79.6	79.8	80.0	80.3	80.5	80.7	80.9	81.1	81.3	81.5	81.7	82.0	82.2
.5	76.9	77.1	77.3	77.6	77.9	78.1	78.3	78.7	78.9	79.1	79.3	79.6	79.8	80.0	80.2	80.4	80.6	80.8	81.0	81.3	81.4
15.0	76.0	76.3	76.5	76.8	77.1	77.3	77.5	77.8	78.1	78.3	78.5	78.8	79.0	79.2	79.4	79.6	79.8	80.1	80.3	80.5	80.7
.5	75.2	75.5	75.7	76.0	76.3	76.5	76.7	77.0	77.3	77.5	77.7	78.1	78.3	78.5	78.7	78.9	79.1	79.4	79.6	79.8	80.0
16.0	74.3	74.6	74.9	75.2	75.4	75.6	75.9	76.2	76.5	76.8	77.0	77.3	77.5	77.8	78.0	78.2	78.4	78.7	78.9	79.1	79.3
.5	73.5	73.8	74.1	74.4	74.6	74.9	75.1	75.4	75.7	76.0	76.3	76.5	76.8	77.1	77.3	77.5	77.7	78.0	78.2	78.4	78.6
17.0	72.6	72.9	73.3	73.6	73.8	74.0	74.3	74.6	74.9	75.2	75.5	75.7	76.0	76.3	76.5	76.8	77.0	77.3	77.5	77.7	77.9
.5	71.8	72.1	72.4	72.7	73.0	73.3	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.6	75.8	76.1	76.3	76.6	76.8	77.0	77.2
18.0	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.8	75.1	75.4	75.6	75.8	76.0	76.3	76.5
.5	70.0	70.3	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.5	73.7	74.1	74.4	74.6	74.9	75.1	75.3	75.6	75.8
19.0	69.1	69.5	69.8	70.1	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.7	72.9	73.3	73.6	73.9	74.2	74.4	74.6	74.9	75.1
.5	68.2	68.6	68.9	69.3	69.6	69.9	70.3	70.5	70.9	71.2	71.5	71.9	72.2	72.6	72.9	73.1	73.4	73.7	73.9	74.2	74.4
20.0	67.3	67.7	68.0	68.4	68.8	69.0	69.4	69.7	70.1	70.5	70.8	71.1	71.4	71.8	72.1	72.4	72.7	73.0	73.2	73.5	73.7
.5	66.4	66.8	67.2	67.6	68.1	68.3	68.5	68.9	69.3	69.7	70.0	70.3	70.6	71.0	71.3	71.6	71.9	72.2	72.4	72.7	73.0
21.0	65.5	65.9	66.3	66.8	67.3	67.5	67.8	68.2	68.5	68.8	69.2	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3
.5	64.6	65.0	65.4	65.9	66.5	66.8	67.1	67.4	67.7	68.0	68.4	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5

TABLE 27  
CONDENSER-WATER  
Weight per unit weight of water evaporated from juice  
R. S. Norris' tables extended \*  
FOR SINGLE EFFECTS (VACUUM-PANS)

Temperature ° C. of Infection-water	Temperature of Water Coming Out of Condenser in ° C.														
	30	32	34	36	38	40	42	44	46	48	50	52	54	56	60
5	24.7	22.8	21.3	19.9	18.7	17.6	16.7	15.8	15.0	14.3	13.7	13.1	12.6	12.1	11.6
6	25.6	23.7	22.0	20.0	19.2	18.1	17.1	16.2	15.4	14.7	14.0	13.4	12.8	12.3	11.8
7	26.7	24.6	22.8	21.2	19.8	18.6	17.6	16.6	15.8	15.0	14.3	13.7	13.1	12.5	12.0
8	27.9	25.6	23.6	21.9	20.4	19.2	18.0	17.0	16.1	15.3	14.6	13.9	13.3	12.8	12.3
9	29.2	26.8	24.5	22.7	21.1	19.8	18.6	17.5	16.6	15.7	14.9	14.2	13.6	13.0	12.5
10	30.6	27.8	25.5	23.5	21.8	20.4	19.1	18.0	17.0	16.1	15.3	14.6	13.9	13.3	12.7
11	32.1	29.1	26.5	24.4	22.6	21.0	19.7	18.5	17.4	16.5	15.7	14.9	14.2	13.6	13.0
12	33.9	30.5	27.7	25.4	23.4	21.8	20.3	19.0	17.9	16.9	16.0	15.2	14.5	13.9	13.3
13	35.8	32.0	29.0	26.5	24.3	22.5	21.0	19.6	18.4	17.4	16.4	15.6	14.8	14.2	13.5
14	38.0	33.7	30.4	27.6	25.3	23.4	21.7	20.2	19.0	17.9	16.9	16.0	15.2	14.5	13.8
15	40.4	35.7	31.9	28.9	26.4	24.2	22.5	20.9	19.6	18.4	17.3	16.4	15.6	14.8	14.1
16	43.2	37.8	33.6	30.3	27.5	25.2	23.3	21.6	20.2	18.9	17.8	16.8	15.9	15.1	14.4
17	46.5	40.3	35.6	31.8	28.8	26.3	24.2	22.4	20.8	19.5	18.3	17.3	16.3	15.5	14.7
18	50.3	43.1	37.8	33.5	30.2	27.4	25.1	23.2	21.6	20.1	18.9	17.7	16.8	15.9	15.1
19	54.8	46.3	40.2	35.4	31.7	28.7	26.2	24.1	22.3	20.8	19.4	18.3	17.2	16.3	15.4
20	60.1	50.1	43.0	37.6	33.4	30.1	27.3	25.1	23.1	21.5	20.0	18.8	17.7	16.7	15.8
21	66.7	54.6	46.2	40.0	35.3	31.6	28.6	26.1	24.0	22.2	20.7	19.3	18.2	17.2	16.2
22	74.9	59.9	49.9	42.8	37.5	33.3	30.0	27.2	25.0	23.1	21.4	20.0	18.7	17.6	16.7
23	85.5	66.5	54.4	46.0	39.9	35.2	31.5	28.5	26.0	23.9	22.2	20.6	19.3	18.1	17.1
24	99.6	74.7	59.7	49.8	42.7	37.3	33.2	29.9	27.2	24.9	23.0	21.3	19.9	18.7	17.6
25	119.3	85.2	66.3	53.2	45.9	39.8	35.1	31.4	28.4	25.9	23.9	22.1	20.6	19.2	18.1
26	148.8	98.2	74.4	59.5	49.6	42.5	37.2	33.1	29.8	27.1	24.8	22.9	21.3	19.8	18.6
27	198.1	118.9	84.9	65.8	54.0	45.7	39.6	35.0	31.3	28.3	25.8	23.8	22.0	20.5	19.2
28	.....	148.3	98.9	74.2	59.3	49.4	42.4	37.1	33.0	29.7	27.0	24.7	22.8	21.2	19.8
29	.....	197.4	118.5	84.6	65.6	53.9	45.6	39.5	34.8	31.2	28.2	25.8	23.7	21.9	20.4

30	.....	147.8	98.6	73.9	59.1	49.3	42.2	37.0	32.9	29.6	26.9	24.6	22.7	21.1	19.7
31	.....	195.8	118.1	84.4	65.6	53.7	45.4	39.3	34.7	31.1	28.2	25.7	23.6	21.9	20.3
32	.....	.....	147.3	98.2	73.7	58.9	49.2	42.1	36.8	32.7	29.5	26.8	24.5	23.7	21.0
33	.....	.....	196.1	117.6	84.1	65.2	53.4	45.3	39.2	34.6	31.0	28.0	25.6	23.5	21.8
34	.....	.....	.....	146.8	97.9	73.4	58.7	48.9	41.9	36.7	32.6	29.4	26.7	24.4	22.6
35	.....	.....	.....	195.6	117.2	83.8	65.1	53.3	45.1	39.1	34.5	30.9	27.9	25.5	23.4
36	.....	.....	.....	.....	146.2	97.6	73.2	58.5	48.7	41.8	36.6	32.5	29.3	26.8	24.4
37	.....	.....	.....	.....	194.8	116.8	83.5	64.9	53.1	44.9	38.9	34.4	30.7	27.8	25.4
38	.....	.....	.....	.....	.....	145.8	97.2	72.9	58.3	48.6	41.7	36.5	32.4	29.2	26.5
39	.....	.....	.....	.....	.....	194.1	116.5	83.2	64.7	52.9	44.8	38.8	34.2	30.6	27.7
40	.....	.....	.....	.....	.....	.....	145.3	96.9	72.7	58.1	48.5	41.5	36.6	32.3	29.1
41	.....	.....	.....	.....	.....	.....	193.5	116.0	82.9	64.5	52.8	44.6	38.7	34.1	30.5
42	.....	.....	.....	.....	.....	.....	144.7	96.5	72.4	57.9	48.3	41.4	36.2	32.2	28.9
43	.....	.....	.....	.....	.....	.....	192.7	115.5	82.7	64.3	52.6	44.5	38.5	34.0	30.4
44	.....	.....	.....	.....	.....	.....	144.3	96.3	72.2	57.7	48.1	41.2	36.1	31.9	28.6
45	.....	.....	.....	.....	.....	.....	192.1	115.3	82.4	64.1	52.4	44.3	38.4	33.9	30.3
46	.....	.....	.....	.....	.....	.....	143.8	96.0	71.9	57.5	47.9	41.1	36.0	31.8	28.5
47	.....	.....	.....	.....	.....	.....	191.4	114.9	82.1	63.8	52.2	44.2	38.3	33.8	30.2
48	.....	.....	.....	.....	.....	.....	.....	143.4	95.6	71.7	57.3	47.8	41.0	36.5	30.7
49	.....	.....	.....	.....	.....	.....	.....	190.8	114.5	81.4	63.6	52.0	44.0	38.1	30.1
50	.....	.....	.....	.....	.....	.....	.....	.....	142.9	95.2	71.4	57.1	46.5	39.4	30.6
51	.....	.....	.....	.....	.....	.....	.....	.....	190.1	114.0	81.5	63.4	51.9	46.9	30.9
52	.....	.....	.....	.....	.....	.....	.....	.....	142.2	94.9	71.2	56.8	46.2	39.1	30.8
53	.....	.....	.....	.....	.....	.....	.....	.....	189.4	113.5	81.2	63.1	51.2	46.2	30.8
54	.....	.....	.....	.....	.....	.....	.....	.....	189.4	113.5	81.2	63.1	51.2	46.2	30.8

## CALCULATION OF THE CONDENSER-WATER

To calculate the condenser-water ( $W$ ) for other pressures than 25 inches (basis of the tables) and for other evaporator combinations than single-, triple- and quadruple-effect, use the following formula, factors and constants:

$$W = \frac{\text{Constant} - T, \text{ } ^\circ \text{C. of condenser-water}}{T, \text{ } ^\circ \text{C. of condenser-water} - T, \text{ } ^\circ \text{C. of injection-water}} + 1.$$

The value of  $W$  must be multiplied by the factor appropriate to the evaporator combination. (Continued on p. 445.)



30	.....	.....	54.7	36.5	27.4	21.9	18.2	15.6	13.7	12.2	11.0	10.0	9.1	8.4	7.8	7.2
31	.....	.....	72.9	43.7	31.2	24.3	19.9	16.8	14.5	12.8	11.5	10.4	9.5	8.7	8.1	7.5
32	.....	.....	.....	54.9	36.3	27.3	21.8	18.2	15.6	13.6	12.1	10.9	9.9	9.1	8.4	7.8
33	.....	.....	.....	72.6	43.5	31.1	24.1	19.8	16.8	14.5	12.8	11.5	10.3	9.5	8.7	8.1
34	.....	.....	.....	.....	54.3	36.2	27.1	21.7	18.1	15.3	13.6	12.0	10.9	9.9	9.0	8.4
35	.....	.....	.....	.....	72.3	43.4	30.9	24.1	19.7	16.8	14.5	12.7	11.4	10.3	9.4	8.7
36	.....	.....	.....	.....	.....	54.1	36.1	27.1	21.6	18.0	15.4	13.5	12.0	10.8	9.8	9.0
37	.....	.....	.....	.....	.....	72.1	43.3	30.9	24.0	19.6	16.6	14.4	12.7	11.3	10.3	9.4
38	.....	.....	.....	.....	.....	.....	53.9	35.9	27.0	21.5	18.0	15.4	13.5	11.9	10.8	9.8
39	.....	.....	.....	.....	.....	.....	71.8	43.1	30.8	23.9	19.5	16.5	14.3	12.6	11.3	10.3
40	.....	.....	.....	.....	.....	.....	.....	53.8	35.8	26.9	21.5	17.9	15.3	13.5	11.9	10.8
41	.....	.....	.....	.....	.....	.....	.....	71.6	42.9	30.7	23.8	19.5	16.5	14.3	12.6	11.2
42	.....	.....	.....	.....	.....	.....	.....	.....	53.6	35.7	26.8	21.4	17.8	15.3	13.4	11.8
43	.....	.....	.....	.....	.....	.....	.....	.....	71.4	42.8	30.6	23.8	19.5	16.5	14.2	12.5
44	.....	.....	.....	.....	.....	.....	.....	.....	.....	53.4	35.6	26.7	21.3	17.8	15.2	13.3
45	.....	.....	.....	.....	.....	.....	.....	.....	.....	71.1	42.7	30.5	23.7	19.4	16.4	14.2
46	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	53.3	35.5	26.6	21.2	17.7	15.1
47	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	70.9	42.5	30.4	23.6	19.3	16.3
48	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	53.1	35.3	26.5	21.2	17.6
49	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	70.7	42.4	30.3	23.5	19.2
50	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
51	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
52	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
53	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
54	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....

## METHOD OF USING NORRIS' TABLES

Calculate the evaporation from clarified juice to sirup ( $E$ ) by the following formula, in which  $B$  is the Brix of the juice and  $b$  that of the sirup:

$$E = (B - b) \div B \times 100, \text{ in terms of the juice}$$

The next stages of the calculations are best illustrated by an example in quadruple-effect evaporation (continued on page 447):

## Factors

Single effect	.....1.000
Double effect	.....0.534
Triple effect	.....0.370
Quadruple effect	.....0.284
Quintuple effect	.....0.235

TABLE 27—Continued  
CONDENSER-WATER  
FOR QUADRUPLER EFFECTS

Temperature ° C. of Infection-water	Temperature of Water Coming Out of Condenser in ° C.											
	30	32	34	36	38	40	42	44	46	48	50	52
5	7.0	6.5	6.0	5.6	5.3	5.0	4.7	4.5	4.3	4.1	3.9	3.7
6	7.3	6.7	6.2	5.8	5.5	5.1	4.9	4.6	4.4	4.2	4.0	3.8
7	7.6	7.0	6.5	6.0	5.6	5.3	5.0	4.7	4.5	4.3	4.1	3.9
8	7.9	7.3	6.7	6.2	5.8	5.4	5.1	4.8	4.6	4.4	4.1	4.0
9	8.3	7.6	7.0	6.4	6.0	5.6	5.3	5.0	4.7	4.5	4.2	4.0
10	8.7	7.9	7.2	6.7	6.2	5.8	5.4	5.1	4.8	4.6	4.3	4.1
11	9.1	8.3	7.5	6.9	6.4	6.0	5.6	5.3	5.0	4.7	4.4	4.2
12	9.3	8.7	7.9	7.2	6.7	6.2	5.8	5.4	5.1	4.8	4.6	4.3
13	10.2	9.1	8.2	7.5	6.9	6.4	6.0	5.6	5.2	4.9	4.7	4.4
14	10.8	9.6	8.6	7.8	7.2	6.6	6.2	5.7	5.4	5.1	4.8	4.5
15	11.5	10.1	9.1	8.2	7.5	6.9	6.4	5.9	5.6	5.2	4.9	4.7
16	12.3	10.7	9.6	8.6	7.8	7.2	6.6	6.1	5.7	5.4	5.1	4.8
17	13.2	11.4	10.1	9.0	8.2	7.5	6.9	6.4	5.9	5.5	5.2	4.9
18	14.3	12.2	10.7	9.5	8.6	7.8	7.1	6.6	6.1	5.7	5.4	5.0
19	15.6	13.2	11.4	10.1	9.0	8.1	7.4	6.8	6.3	5.9	5.5	5.2
20	17.1	14.2	12.2	10.7	9.5	8.5	7.8	7.1	6.6	6.1	5.7	5.3
21	18.9	15.5	13.1	11.4	10.0	9.0	8.1	7.4	6.8	6.3	5.9	5.5
22	21.3	17.0	14.2	12.2	10.6	9.5	8.5	7.7	7.1	6.5	6.1	5.7
23	24.3	18.9	15.4	13.1	11.3	10.0	8.9	8.1	7.4	6.8	6.3	5.9
24	28.3	21.2	17.0	14.1	12.1	10.6	9.4	8.5	7.7	7.1	6.5	6.1
25	33.9	24.2	18.8	15.4	13.0	11.3	10.0	8.9	8.1	7.4	6.8	6.3
26	42.3	28.2	21.1	16.9	14.1	12.1	10.6	9.4	8.5	7.7	7.0	6.5
27	56.3	33.8	24.1	18.7	15.3	13.0	11.3	9.9	8.9	8.0	7.3	6.8
28	.....	42.2	28.1	21.1	16.9	14.0	12.0	10.5	9.4	8.4	7.7	7.0
29	.....	.....	33.6	24.0	18.6	15.3	12.9	11.2	9.9	8.9	8.0	7.3
60	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
58	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
56	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
54	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
52	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
50	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
48	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
46	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
44	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
42	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
40	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
38	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
36	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
34	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
32	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3
30	3.2	3.3	3.4	3.5	3.6	3.7	3.8	3.9	4.0	4.1	4.2	4.3



30	.....	42.0	28.0	21.0	16.8	14.0	12.0	10.5	9.3	8.4	7.6	7.0	6.5	6.0	5.6
31	.....	56.0	33.6	24.0	18.6	15.3	12.9	11.1	9.9	8.8	8.0	7.3	6.7	6.2	5.8
32	.....	.....	41.9	27.9	20.9	16.7	13.9	11.9	10.4	9.3	8.4	7.9	7.3	6.7	6.2
33	.....	.....	55.3	33.4	23.9	18.5	15.2	12.8	11.1	9.8	8.8	8.0	7.3	6.7	6.2
34	.....	.....	.....	41.7	27.9	20.8	16.7	13.9	11.9	10.4	9.3	8.4	7.6	7.0	6.5
35	.....	.....	.....	55.3	33.3	23.7	18.5	15.1	12.8	11.1	9.8	8.8	7.9	7.3	6.7
36	.....	.....	.....	41.5	27.7	20.8	16.6	13.8	11.9	10.4	9.2	8.3	7.6	7.0	6.5
37	.....	.....	.....	55.2	33.2	23.7	18.4	15.1	12.7	11.0	9.8	8.7	7.9	7.3	6.7
38	.....	.....	.....	41.4	27.6	20.7	16.5	13.8	11.8	10.4	9.2	8.3	7.6	7.0	6.5
39	.....	.....	.....	55.1	33.1	23.6	18.4	15.0	12.7	11.0	9.7	8.7	7.9	7.3	6.7
40	.....	.....	.....	.....	41.4	27.5	20.5	16.5	13.8	11.8	10.4	9.2	8.3	7.6	7.0
41	.....	.....	.....	.....	55.0	32.9	23.5	18.3	15.0	12.6	11.0	9.7	8.7	7.9	7.3
42	.....	.....	.....	.....	41.1	27.4	20.5	16.4	13.7	11.9	10.3	9.2	8.3	7.6	7.0
43	Weight of mixed juice.....	1500 tons	.....	.....	54.8	32.8	23.5	18.2	14.9	12.6	10.9	9.7	8.7	7.9	7.3
44	Temperature of injection-water....	35° C.	.....	.....	41.0	27.3	20.5	16.4	13.6	11.7	10.2	9.2	8.3	7.6	7.0
45	Temperature of condenser-water....	50° C.	.....	.....	54.6	32.7	23.4	18.2	14.9	12.6	10.8	9.7	8.7	7.9	7.3
46	Brix of the juice.....	15	.....	.....	40.9	27.2	20.4	16.3	13.6	11.7	10.2	9.2	8.3	7.6	7.0
47	Brix of the sirup.....	60	.....	.....	54.4	32.6	23.3	18.1	14.8	12.5	10.8	9.7	8.7	7.9	7.3
48	.....	.....	.....	.....	.....	40.7	27.1	20.3	16.3	13.5	11.7	10.2	9.2	8.3	7.6
49	$E = [(60 - 15) \div 60] \times 100 = 75$ per cent of weight of juice; $1500 \times .75 = 1125$ tons of water evaporated; referring to the third section of the table, opposite 35° and under 50° we have 11.1, the condenser water per unit of water evaporated; $1125 \times 11.1 = 12,488$ tons of condenser-water.	.....	.....	.....	.....	54.2	32.5	23.1	18.0	14.7	12.5	10.8	9.7	8.7	7.9
50	.....	.....	.....	.....	.....	.....	40.6	27.0	20.2	16.2	14.0	12.5	10.8	9.7	7.9
51	.....	.....	.....	.....	.....	.....	54.0	32.4	23.1	18.0	14.7	12.5	10.8	9.7	7.9
52	.....	.....	.....	.....	.....	.....	.....	40.4	26.9	20.2	16.2	14.0	12.5	10.8	9.7
53	.....	.....	.....	.....	.....	.....	.....	53.8	32.2	23.0	18.0	14.7	12.5	10.8	9.7
54	.....	.....	.....	.....	.....	.....	.....	.....	40.3	26.8	18.0	14.7	12.5	10.8	9.7

TABLE 28

FOR THE REDUCTION OF THE WEIGHT OR VOLUME OF A SIRUP OF A GIVEN DEGREE  
BRIX OR BAUMÉ TO A SIRUP OF 54.3° BRIX OR 30° BAUMÉ.—(SPENCER)

(See Formula Sec. 391)

Initial Density		Equivalent Sirup of 54.3° Brix or 30° Baumé		Initial Density		Equivalent Sirup of 54.3° Brix or 30° Baumé	
Degrees Brix	Degrees Baumé	Per Cents by Weight	Per Cents by Volume	Degrees Brix	Degrees Baumé	Per Cents by Weight	Per Cents by Volume
<b>35.0</b>	19.6	64.46	59.19	<b>39.0</b>	21.8	71.82	67.10
.1	19.65	64.64	59.38	.1	21.8	72.00	67.30
.2	19.7	64.83	59.58	.2	21.9	72.19	67.51
.3	19.8	65.01	59.77	.3	21.9	72.37	67.71
.4	19.8	65.19	59.96	.4	22.0	72.55	67.91
.5	19.9	65.38	60.16	.5	22.05	72.74	68.12
.6	19.9	65.56	60.36	.6	22.1	72.92	68.32
.7	20.0	65.74	60.56	.7	22.2	73.10	68.52
.8	20.0	65.93	60.75	.8	22.2	73.29	68.72
.9	20.1	66.11	60.94	.9	22.3	73.47	68.92
<b>36.0</b>	20.1	66.30	61.14	<b>40.0</b>	22.3	73.66	69.12
.1	20.2	66.48	61.33	.1	22.4	73.84	69.32
.2	20.25	66.67	61.53	.2	22.4	74.02	69.52
.3	20.3	66.85	61.72	.3	22.5	74.21	69.73
.4	20.4	67.03	61.92	.4	22.5	74.40	69.93
.5	20.4	67.22	62.12	.5	22.6	74.58	70.14
.6	20.5	67.40	62.31	.6	22.6	74.76	70.34
.7	20.5	67.59	62.50	.7	22.7	74.94	70.54
.8	20.6	67.77	62.70	.8	22.8	75.13	70.74
.9	20.6	67.95	62.91	.9	22.8	75.31	70.94
<b>37.0</b>	20.7	68.14	63.11	<b>41.0</b>	22.9	75.50	71.15
.1	20.7	68.32	63.31	.1	22.9	75.68	71.35
.2	20.8	68.50	63.51	.2	23.0	75.87	71.55
.3	20.9	68.69	63.70	.3	23.0	76.06	71.75
.4	20.9	68.87	63.90	.4	23.1	76.24	71.95
.5	21.0	69.06	64.10	.5	23.1	76.42	72.16
.6	21.0	69.24	64.30	.6	23.2	76.60	72.37
.7	21.1	69.42	64.49	.7	23.25	76.78	72.58
.8	21.1	69.61	64.69	.8	23.3	76.97	72.79
.9	21.2	69.79	64.89	.9	23.4	77.16	73.00
<b>38.0</b>	21.2	69.98	65.09	<b>42.0</b>	23.4	77.34	73.21
.1	21.3	70.16	65.29	.1	23.5	77.52	73.41
.2	21.35	70.34	65.49	.2	23.5	77.70	73.61
.3	21.4	70.53	65.69	.3	23.6	77.89	73.81
.4	21.5	70.72	65.90	.4	23.6	78.08	74.01
.5	21.5	70.90	66.10	.5	23.7	78.26	74.22
.6	21.6	71.08	66.30	.6	23.7	78.44	74.43
.7	21.6	71.26	66.50	.7	23.8	78.62	74.64
.8	21.7	71.45	66.70	.8	23.8	78.81	74.86
.9	21.7	71.63	66.90	.9	23.9	79.00	75.08

TABLE 28—Continued

TABLE FOR THE REDUCTION OF THE WEIGHT OR VOLUME OF A SIRUP, ETC.

Initial Density		Equivalent Sirup of 54.3° Brix or 30° Baumé		Initial Density		Equivalent Sirup of 54.3° Brix or 30° Baumé	
Degrees Brix	Degrees Baumé	Per Cents by Weight	Per Cents by Volume	Degrees Brix	Degrees Baumé	Per Cents by Weight	Per Cents by Volume
43.0	23.95	79.19	75.29	47.0	26.1	86.55	83.76
.1	24.0	79.37	75.49	.1	26.2	86.73	83.97
.2	24.1	79.55	75.69	.2	26.2	86.91	84.18
.3	24.1	79.74	75.89	.3	26.3	87.10	84.39
.4	24.2	79.92	76.10	.4	26.3	87.29	84.60
.5	24.2	80.11	76.31	.5	26.4	87.47	84.82
.6	24.3	80.29	76.52	.6	26.4	87.65	85.04
.7	24.3	80.47	76.73	.7	26.5	87.83	85.26
.8	24.4	80.66	76.95	.8	26.5	88.02	85.48
.9	24.4	80.85	77.17	.9	26.6	88.21	85.70
44.0	24.5	81.03	77.38	48.0	26.6	88.39	85.92
.1	24.55	81.21	77.59	.1	26.7	88.57	86.13
.2	24.6	81.39	77.80	.2	26.75	88.75	86.35
.3	24.65	81.58	78.01	.3	26.8	88.94	86.57
.4	24.7	81.77	78.22	.4	26.9	89.13	86.79
.5	24.8	81.95	78.43	.5	26.9	89.31	87.01
.6	24.8	82.13	78.64	.6	27.0	89.49	87.23
.7	24.9	82.31	78.85	.7	27.0	89.67	87.45
.8	24.9	82.50	79.06	.8	27.1	89.86	87.67
.9	25.0	82.69	79.27	.9	27.1	90.15	87.89
45.0	25.0	82.87	79.49	49.0	27.2	90.24	88.11
.1	25.1	83.05	79.70	.1	27.2	90.42	88.33
.2	25.1	83.23	79.91	.2	27.3	90.60	88.55
.3	25.2	83.42	80.12	.3	27.3	90.78	88.77
.4	25.2	83.61	80.33	.4	27.4	90.96	88.99
.5	25.3	83.79	80.54	.5	27.4	91.16	89.21
.6	25.4	83.97	80.75	.6	27.5	91.35	89.43
.7	25.4	84.15	80.96	.7	27.6	91.54	89.65
.8	25.5	84.34	81.18	.8	27.6	91.72	89.87
.9	25.5	84.53	81.40	.9	27.7	91.90	90.09
46.0	25.6	84.71	81.61	50.0	27.7	92.08	90.31
.1	25.6	84.89	81.82	.1	27.8	92.26	90.53
.2	25.7	85.07	82.03	.2	27.8	92.45	90.75
.3	25.7	85.26	82.24	.3	27.9	92.63	90.97
.4	25.8	85.45	82.45	.4	27.9	92.82	91.19
.5	25.8	85.63	82.66	.5	28.0	93.00	91.41
.6	25.9	85.81	82.87	.6	28.0	93.19	91.63
.7	25.95	85.99	83.09	.7	28.1	93.37	91.85
.8	26.0	86.18	83.31	.8	28.1	93.55	92.07
.9	26.1	86.37	83.53	.9	28.2	93.73	92.30

TABLE 28—Continued

TABLE FOR THE REDUCTION OF THE WEIGHT OR VOLUME OF A SIRUP, ETC.

Initial Density		Equivalent Sirup of 54.3° Brix or 30° Baumé		Initial Density		Equivalent Sirup of 54.3° Brix or 30° Baumé	
Degrees Brix	Degrees Baumé	Per Cents by Weight	Per Cents by Volume	Degrees Brix	Degrees Baumé	Per Cents by Weight	Per Cents by Volume
<b>51.0</b>	28.2	93.92	92.53	<b>56.0</b>	30.9	103.13	103.92
.1	28.3	94.10	92.75	.1	30.9	103.31	104.15
.2	28.35	94.29	92.97	.2	31.0	103.49	104.38
.3	28.4	94.47	93.19	.3	31.05	103.68	104.61
.4	28.5	94.65	93.41	.4	31.1	103.86	104.84
.5	28.5	94.84	93.63	.5	31.2	104.05	105.07
.6	28.6	95.02	93.85	.6	31.2	104.23	105.30
.7	28.6	95.20	94.07	.7	31.3	104.41	105.54
.8	28.7	95.39	94.30	.8	31.3	104.60	105.78
.9	28.7	95.58	94.53	.9	31.4	104.78	106.02
<b>52.0</b>	28.3	95.76	94.77	<b>57.0</b>	31.4	104.97	106.26
.1	28.8	95.94	94.99	.1	31.5	105.15	106.49
.2	28.9	96.13	95.21	.2	31.5	105.34	106.72
.3	28.9	96.31	95.43	.3	31.6	105.52	106.95
.4	29.0	96.50	95.65	.4	31.6	105.70	107.18
.5	29.0	96.68	95.87	.5	31.7	105.89	107.41
.6	29.1	96.87	96.09	.6	31.7	106.07	107.65
.7	29.15	97.05	96.32	.7	31.8	106.25	107.89
.8	29.2	97.23	96.55	.8	31.8	106.44	108.13
.9	29.2	97.42	96.79	.9	31.9	106.62	108.37
<b>53.0</b>	29.3	97.60	97.02	<b>58.0</b>	31.9	106.81	108.61
.1	29.4	97.79	97.25	.1	32.0	106.99	108.84
.2	29.4	97.98	97.48	.2	32.0	107.17	109.08
.3	29.5	98.16	97.71	.3	32.1	107.35	109.32
.4	29.5	98.34	97.94	.4	32.15	107.54	109.56
.5	29.6	98.52	98.17	.5	32.2	107.73	109.80
.6	29.6	98.70	98.40	.6	32.3	107.91	110.04
.7	29.7	98.89	98.63	.7	32.3	108.09	110.28
.8	29.7	99.07	98.86	.8	32.4	108.28	110.52
.9	29.8	99.26	99.08	.9	32.4	108.47	110.76
<b>54.0</b>	29.8	99.44	99.30	<b>59.0</b>	32.5	108.65	111.00
.1	29.9	99.62	99.53	.1	32.5	108.83	111.23
.2	29.9	99.81	99.76	.2	32.6	109.02	111.47
.3	30.0	100.00	100.00	.3	32.6	109.20	111.71
.4	30.05	100.18	100.22	.4	32.7	109.38	111.95
.5	30.1	100.36	100.45	.5	32.7	109.56	112.19
.6	30.2	100.55	100.68	.6	32.8	109.75	112.43
.7	30.2	100.73	100.91	.7	32.8	109.93	112.67
.8	30.3	100.91	101.14	.8	32.9	110.12	112.91
.9	30.3	101.09	101.37	.9	32.9	110.30	113.15
<b>55.0</b>	30.4	101.28	101.61	<b>60.0</b>	33.0	110.49	113.39
.1	30.4	101.46	101.84	.1	33.0	110.68	113.63
.2	30.5	101.64	102.07	.2	33.1	110.86	113.87
.3	30.5	101.83	102.30	.3	33.1	111.04	114.11
.4	30.6	102.01	102.53	.4	33.2	111.23	114.35
.5	30.6	102.20	102.76	.5	33.2	111.41	114.59
.6	30.7	102.38	102.99	.6	33.3	111.60	114.83
.7	30.7	102.56	103.22	.7	33.35	111.78	114.97
.8	30.8	102.75	103.45	.8	33.4	111.96	115.21
.9	30.8	102.94	103.68	.9	33.45	112.14	115.45

TABLE 29

SHOWING A COMPARISON OF THE DEGREES BRIX AND BAUMÉ, AND OF THE SPECIFIC GRAVITY OF SUGAR SOLUTIONS AT 17 1/4° C.—(STAMMER)<sup>1</sup>

Degree Brix (Per Cent Sugar)	Degree Baumé (Corrected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Corrected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Corrected)	Specific Gravity
0.0	0.0	1.00000	3.0	1.7	1.01173	6.0	3.4	1.02373
.1	0.1	1.00038	.1	1.8	1.01213	.1	3.5	1.02413
.2	0.1	1.00077	.2	1.8	1.01252	.2	3.5	1.02454
.3	0.2	1.00116	.3	1.9	1.01292	.3	3.6	1.02494
.4	0.2	1.00155	.4	1.9	1.01332	.4	3.6	1.02535
.5	0.3	1.00193	.5	2.0	1.01371	.5	3.7	1.02575
.6	0.3	1.00232	.6	2.0	1.01411	.6	3.7	1.02616
.7	0.4	1.00271	.7	2.1	1.01451	.7	3.8	1.02657
.8	0.45	1.00310	.8	2.2	1.01491	.8	3.9	1.02694
.9	0.5	1.00349	.9	2.2	1.01531	.9	3.9	1.02738
1.0	0.6	1.00388	4.0	2.3	1.01570	7.0	4.0	1.02779
.1	0.6	1.00427	.1	2.3	1.01610	.1	4.0	1.02819
.2	0.7	1.00466	.2	2.4	1.01650	.2	4.1	1.02860
.3	0.7	1.00505	.3	2.4	1.01690	.3	4.1	1.02901
.4	0.8	1.00544	.4	2.5	1.01730	.4	4.2	1.02942
.5	0.85	1.00583	.5	2.55	1.01770	.5	4.25	1.02983
.6	0.9	1.00622	.6	2.6	1.01810	.6	4.3	1.03024
.7	1.0	1.00662	.7	2.7	1.01850	.7	4.4	1.03064
.8	1.0	1.00701	.8	2.7	1.01890	.8	4.4	1.03105
.9	1.1	1.00740	.9	2.8	1.01930	.9	4.5	1.03146
2.0	1.1	1.00779	5.0	2.8	1.01970	8.0	4.5	1.03187
.1	1.2	1.00818	.1	2.9	1.02010	.1	4.6	1.03228
.2	1.2	1.00858	.2	2.95	1.02051	.2	4.6	1.03270
.3	1.3	1.00897	.3	3.0	1.02091	.3	4.7	1.03311
.4	1.4	1.00936	.4	3.1	1.02131	.4	4.8	1.03352
.5	1.4	1.00976	.5	3.1	1.02171	.5	4.8	1.03393
.6	1.5	1.01015	.6	3.2	1.02211	.6	4.9	1.03434
.7	1.5	1.01055	.7	3.2	1.02252	.7	4.9	1.03475
.8	1.6	1.01094	.8	3.3	1.02292	.8	5.0	1.03517
.9	1.6	1.01134	.9	3.35	1.02333	.9	5.0	1.03558

## CORRECTION FOR TEMPERATURE, BRIX SPINDLE.—(GERLACH)

Temp. °C.	Temp. °F.	Approximate Degree Brix and Correction				
		0	5	10	15	
13	55.4	.14	.18	.19	.21	Subtract.
14	57.2	.12	.15	.16	.17	
15	59.	.09	.11	.12	.14	
16	60.8	.06	.07	.08	.09	
17	62.6	.02	.02	.03	.03	
18	64.4	.02	.03	.03	.03	Add.
19	66.2	.06	.08	.08	.09	
20	68.	.11	.14	.15	.17	
21	69.8	.16	.20	.22	.24	
22	71.6	.21	.26	.29	.31	
23	73.4	.27	.32	.35	.37	
24	75.2	.32	.38	.41	.43	
25	77.	.37	.44	.47	.49	

<sup>1</sup>The degrees Baumé of this table are the so-called corrected or "new" degrees according to Gerlach (modulus 146.78) see Sec. 242.

TABLE 29—Continued

SHOWING A COMPARISON OF THE DEGREES BRIX AND BAUMÉ, ETC., OF SUGAR SOLUTIONS

Degree Brix (Per Cent Sugar)	Degree Baumé (Corrected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Corrected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Corrected)	Specific Gravity
9.0	5.1	1.03599	12.0	6.8	1.04852	15.0	8.5	1.06133
.1	5.2	1.03640	.1	6.8	1.04894	.1	8.5	1.06176
.2	5.2	1.03682	.2	6.9	1.04937	.2	8.55	1.06219
.3	5.3	1.03723	.3	7.0	1.04979	.3	8.6	1.06262
.4	5.3	1.03765	.4	7.0	1.05021	.4	8.7	1.06306
.5	5.4	1.03806	.5	7.1	1.05064	.5	8.8	1.06349
.6	5.4	1.03848	.6	7.1	1.05106	.6	8.8	1.06392
.7	5.5	1.03889	.7	7.2	1.05149	.7	8.9	1.06436
.8	5.55	1.03931	.8	7.2	1.05191	.8	8.9	1.06479
.9	5.6	1.03972	.9	7.3	1.05233	.9	9.0	1.06522
10.0	5.7	1.04014	13.0	7.4	1.05276	16.0	9.0	1.06566
.1	5.7	1.04055	.1	7.4	1.05318	.1	9.1	1.06609
.2	5.8	1.04097	.2	7.5	1.05361	.2	9.2	1.06653
.3	5.8	1.04139	.3	7.5	1.05404	.3	9.2	1.06696
.4	5.9	1.04180	.4	7.6	1.05446	.4	9.3	1.06740
.5	5.9	1.04222	.5	7.6	1.05489	.5	9.3	1.06783
.6	6.0	1.04264	.6	7.7	1.05532	.6	9.4	1.06827
.7	6.1	1.04306	.7	7.75	1.05574	.7	9.4	1.06871
.8	6.1	1.04348	.8	7.8	1.05617	.8	9.5	1.06914
.9	6.2	1.04390	.9	7.9	1.05660	.9	9.5	1.06958
11.0	6.2	1.04431	14.0	7.9	1.05703	17.0	9.6	1.07002
.1	6.3	1.04473	.1	8.0	1.05746	.1	9.7	1.07046
.2	6.3	1.04515	.2	8.0	1.05789	.2	9.7	1.07090
.3	6.4	1.04557	.3	8.1	1.05831	.3	9.8	1.07133
.4	6.5	1.04599	.4	8.1	1.05874	.4	9.8	1.07177
.5	6.5	1.04641	.5	8.2	1.05917	.5	9.9	1.07221
.6	6.6	1.04683	.6	8.3	1.05960	.6	9.9	1.07265
.7	6.6	1.04726	.7	8.3	1.06003	.7	10.0	1.07309
.8	6.7	1.04768	.8	8.4	1.06047	.8	10.0	1.07353
.9	6.7	1.04810	.9	8.4	1.06090	.9	10.1	1.07397

CORRECTION FOR TEMPERATURE, BRIX SPINDLE. (GERLACH)

Temp. ° C.	Temp. ° F.	Approximate Degree Brix and Correction				
		15	20	25	30	
13	55.4	.21	.22	.24	.26	Subtract.
14	57.2	.17	.18	.19	.21	
15	59.	.14	.14	.15	.16	
16	60.8	.09	.10	.10	.11	
17	62.6	.03	.03	.04	.04	
18	64.4	.03	.03	.03	.03	Add.
19	66.2	.09	.09	.10	.10	
20	68.	.17	.17	.18	.18	
21	69.8	.24	.24	.25	.25	
22	71.6	.31	.31	.32	.32	
23	73.4	.37	.38	.39	.39	
24	75.2	.43	.44	.46	.46	
25	77.	.49	.51	.53	.54	

NOTE.—For temperatures above 17  $\frac{1}{2}$ ° C. add the correction to the reading at the observed temperature; below 17  $\frac{1}{2}$ ° C. subtract.

Obtain Baumé corrections from corresponding degree Brix.

TABLE 29—Continued

SHOWING A COMPARISON OF THE DEGREES BRIX AND BAUMÉ, ETC.

Degree Brix (Per Cent Sugar)	Degree Baumé (Cor- rected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Cor- rected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Cor- rected)	Specific Gravity
18.0	10.1	1.07441	23.0	13.0	1.09686	28.0	15.7	1.12013
.1	10.2	1.07485	.1	13.0	1.09732	.1	15.8	1.12060
.2	10.3	1.07530	.2	13.1	1.09777	.2	15.8	1.12107
.3	10.3	1.07574	.3	13.1	1.09823	.3	15.9	1.12155
.4	10.4	1.07618	.4	13.2	1.09869	.4	16.0	1.12202
.5	10.4	1.07662	.5	13.2	1.09915	.5	16.0	1.12250
.6	10.5	1.07706	.6	13.3	1.09961	.6	16.1	1.12297
.7	10.5	1.07751	.7	13.3	1.10007	.7	16.1	1.12345
.8	10.6	1.07795	.8	13.4	1.10053	.8	16.2	1.12393
.9	10.6	1.07839	.9	13.5	1.10099	.9	16.2	1.12440
19.0	10.7	1.07884	24.0	13.5	1.10145	29.0	16.3	1.12488
.1	10.8	1.07928	.1	13.6	1.10191	.1	16.3	1.12536
.2	10.8	1.07973	.2	13.6	1.10237	.2	16.4	1.12583
.3	10.9	1.08017	.3	13.7	1.10283	.3	16.5	1.12631
.4	10.9	1.08062	.4	13.7	1.10329	.4	16.5	1.12679
.5	11.0	1.08106	.5	13.8	1.10375	.5	16.6	1.12727
.6	11.1	1.08151	.6	13.8	1.10421	.6	16.6	1.12775
.7	11.1	1.08196	.7	13.9	1.10468	.7	16.7	1.12823
.8	11.2	1.08240	.8	14.0	1.10514	.8	16.7	1.12871
.9	11.2	1.08285	.9	14.0	1.10560	.9	16.8	1.12919
20.0	11.3	1.08329	25.0	14.1	1.10607	30.0	16.8	1.12967
.1	11.3	1.08374	.1	14.1	1.10653	.1	16.9	1.13015
.2	11.4	1.08419	.2	14.2	1.10700	.2	16.95	1.13063
.3	11.5	1.08464	.3	14.2	1.10746	.3	17.0	1.13111
.4	11.5	1.08509	.4	14.3	1.10793	.4	17.1	1.13159
.5	11.6	1.08553	.5	14.3	1.10839	.5	17.1	1.13207
.6	11.6	1.08599	.6	14.4	1.10886	.6	17.2	1.13255
.7	11.7	1.08643	.7	14.5	1.10932	.7	17.2	1.13304
.8	11.7	1.08688	.8	14.5	1.10979	.8	17.3	1.13352
.9	11.8	1.08733	.9	14.6	1.11026	.9	17.3	1.13400
21.0	11.8	1.08778	26.0	14.6	1.11072	31.0	17.4	1.13449
.1	11.9	1.08824	.1	14.7	1.11119	.1	17.4	1.13497
.2	11.95	1.08869	.2	14.7	1.11166	.2	17.5	1.13545
.3	12.0	1.08914	.3	14.8	1.11213	.3	17.6	1.13594
.4	12.0	1.08959	.4	14.85	1.11259	.4	17.6	1.13642
.5	12.1	1.09004	.5	14.9	1.11306	.5	17.7	1.13691
.6	12.1	1.09049	.6	15.0	1.11353	.6	17.7	1.13740
.7	12.2	1.09095	.7	15.0	1.11400	.7	17.8	1.13788
.8	12.3	1.09140	.8	15.1	1.11447	.8	17.8	1.13837
.9	12.3	1.09185	.9	15.1	1.11494	.9	17.9	1.13885
22.0	12.4	1.09231	27.0	15.2	1.11541	32.0	17.95	1.13934
.1	12.5	1.09276	.1	15.2	1.11588	.1	18.0	1.13983
.2	12.5	1.09321	.2	15.3	1.11635	.2	18.0	1.14032
.3	12.6	1.09367	.3	15.3	1.11682	.3	18.1	1.14081
.4	12.6	1.09412	.4	15.4	1.11729	.4	18.2	1.14129
.5	12.7	1.09458	.5	15.5	1.11776	.5	18.2	1.14178
.6	12.7	1.09503	.6	15.5	1.11824	.6	18.3	1.14227
.7	12.8	1.09549	.7	15.6	1.11871	.7	18.3	1.14276
.8	12.85	1.09595	.8	15.6	1.11918	.8	18.4	1.14325
.9	12.9	1.09640	.9	15.7	1.11965	.9	18.4	1.14374

TABLE 29—Continued

SHOWING A COMPARISON OF THE DEGREES BRIX AND BAUMÉ, ETC.

Degree Brix (Per Cent Sugar)	Degree Baumé (Corrected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Corrected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Corrected)	Specific Gravity
<b>33.0</b>	18.5	1.14423	<b>38.0</b>	21.2	1.16920	<b>43.0</b>	23.95	1.19505
.1	18.55	1.14472	.1	21.3	1.16971	.1	24.0	1.19558
.2	18.6	1.14521	.2	21.35	1.17022	.2	24.1	1.19611
.3	18.7	1.14570	.3	21.4	1.17072	.3	24.1	1.19653
.4	18.7	1.14620	.4	21.5	1.17123	.4	24.2	1.19716
.5	18.8	1.14669	.5	21.5	1.17174	.5	24.2	1.19769
.6	18.8	1.14718	.6	21.6	1.17225	.6	24.3	1.19822
.7	18.9	1.14767	.7	21.6	1.17276	.7	24.3	1.19875
.8	18.9	1.14817	.8	21.7	1.17327	.8	24.4	1.19927
.9	19.0	1.14866	.9	21.7	1.17379	.9	24.4	1.19980
<b>34.0</b>	19.05	1.14915	<b>39.0</b>	21.8	1.17430	<b>44.0</b>	24.5	1.20033
.1	19.1	1.14965	.1	21.8	1.17481	.1	24.55	1.20086
.2	19.2	1.15014	.2	21.9	1.17532	.2	24.6	1.20139
.3	19.2	1.15064	.3	21.9	1.17583	.3	24.65	1.20192
.4	19.3	1.15113	.4	22.0	1.17635	.4	24.7	1.20245
.5	19.3	1.15163	.5	22.05	1.17686	.5	24.8	1.20299
.6	19.4	1.15213	.6	22.1	1.17737	.6	24.8	1.20352
.7	19.4	1.15262	.7	22.2	1.17789	.7	24.9	1.20405
.8	19.5	1.15312	.8	22.2	1.17840	.8	24.9	1.20458
.9	19.5	1.15362	.9	22.3	1.17892	.9	25.0	1.20512
<b>35.0</b>	19.6	1.15411	<b>40.0</b>	22.3	1.17943	<b>45.0</b>	25.0	1.20565
.1	19.65	1.15461	.1	22.4	1.17995	.1	25.1	1.20618
.2	19.7	1.15511	.2	22.4	1.18046	.2	25.1	1.20672
.3	19.8	1.15561	.3	22.5	1.18098	.3	25.2	1.20725
.4	19.8	1.15611	.4	22.5	1.18150	.4	25.2	1.20779
.5	19.9	1.15661	.5	22.6	1.18201	.5	25.3	1.20832
.6	19.9	1.15710	.6	22.6	1.18253	.6	25.4	1.20886
.7	20.0	1.15760	.7	22.7	1.18305	.7	25.4	1.20939
.8	20.0	1.15810	.8	22.8	1.18357	.8	25.5	1.20993
.9	20.1	1.15861	.9	22.8	1.18408	.9	25.5	1.21046
<b>36.0</b>	20.1	1.15911	<b>41.0</b>	22.9	1.18460	<b>46.0</b>	25.6	1.21100
.1	20.2	1.15961	.1	22.9	1.18512	.1	25.6	1.21154
.2	20.25	1.16011	.2	23.0	1.18564	.2	25.7	1.21208
.3	20.3	1.16061	.3	23.0	1.18616	.3	25.7	1.21261
.4	20.4	1.16111	.4	23.1	1.18668	.4	25.8	1.21315
.5	20.4	1.16162	.5	23.1	1.18720	.5	25.8	1.21369
.6	20.5	1.16212	.6	23.2	1.18772	.6	25.9	1.21423
.7	20.5	1.16262	.7	23.25	1.18824	.7	25.95	1.21477
.8	20.6	1.16313	.8	23.3	1.18877	.8	26.0	1.21531
.9	20.6	1.16363	.9	23.4	1.18929	.9	26.1	1.21585
<b>37.0</b>	20.7	1.16413	<b>42.0</b>	23.4	1.18981	<b>47.0</b>	26.1	1.21639
.1	20.7	1.16464	.1	23.5	1.19033	.1	26.2	1.21693
.2	20.8	1.16514	.2	23.5	1.19086	.2	26.2	1.21747
.3	20.9	1.16565	.3	23.6	1.19138	.3	26.3	1.21802
.4	20.9	1.16616	.4	23.6	1.19190	.4	26.3	1.21856
.5	21.0	1.16666	.5	23.7	1.19243	.5	26.4	1.21910
.6	21.0	1.16717	.6	23.7	1.19295	.6	26.4	1.21964
.7	21.1	1.16768	.7	23.8	1.19348	.7	26.5	1.22019
.8	21.1	1.16818	.8	23.8	1.19400	.8	26.5	1.22073
.9	21.2	1.16869	.9	23.9	1.19453	.9	26.6	1.22127



TABLE 29—Continued

SHOWING A COMPARISON OF THE DEGREES BRIX AND BAUMÉ, ETC.

Degree Brix (Per Cent Sugar)	Degree Baumé (Cor- rected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Cor- rected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Cor- rected)	Specific Gravity
48.0	26.6	1.22182	58.0	29.3	1.24951	58.0	31.9	1.27816
.1	26.7	1.22236	.1	29.4	1.25008	.1	32.0	1.27874
.2	26.75	1.22291	.2	29.4	1.25064	.2	32.0	1.27932
.3	26.8	1.22345	.3	29.5	1.25120	.3	32.1	1.27991
.4	26.9	1.22400	.4	29.5	1.25177	.4	32.15	1.28049
.5	26.9	1.22455	.5	29.6	1.25233	.5	32.2	1.28107
.6	27.0	1.22509	.6	29.6	1.25290	.6	32.3	1.28166
.7	27.0	1.22564	.7	29.7	1.25347	.7	32.3	1.28224
.8	27.1	1.22619	.8	29.7	1.25403	.8	32.4	1.28283
.9	27.1	1.22673	.9	29.8	1.25460	.9	32.4	1.28342
49.0	27.2	1.22728	54.0	29.8	1.25517	59.0	32.5	1.28400
.1	27.2	1.22783	.1	29.9	1.25573	.1	32.5	1.28459
.2	27.3	1.22838	.2	29.9	1.25630	.2	32.6	1.28518
.3	27.3	1.22893	.3	30.0	1.25687	.3	32.6	1.28576
.4	27.4	1.22948	.4	30.05	1.25747	.4	32.7	1.28635
.5	27.4	1.23003	.5	30.1	1.25801	.5	32.7	1.28694
.6	27.5	1.23058	.6	30.2	1.25857	.6	32.8	1.28753
.7	27.6	1.23113	.7	30.2	1.25914	.7	32.8	1.28812
.8	27.6	1.23168	.8	30.3	1.25971	.8	32.9	1.28871
.9	27.7	1.23223	.9	30.3	1.26028	.9	32.9	1.28930
50.0	27.7	1.23278	55.0	30.4	1.26086	60.0	33.0	1.28989
.1	27.8	1.23334	.1	30.4	1.26143	.1	33.0	1.29048
.2	27.8	1.23389	.2	30.5	1.26200	.2	33.1	1.29107
.3	27.9	1.23444	.3	30.5	1.26257	.3	33.1	1.29166
.4	27.9	1.23499	.4	30.6	1.26314	.4	33.2	1.29225
.5	28.0	1.23555	.5	30.6	1.26372	.5	33.2	1.29284
.6	28.0	1.23610	.6	30.7	1.26429	.6	33.3	1.29343
.7	28.1	1.23666	.7	30.7	1.26486	.7	33.35	1.29403
.8	28.1	1.23721	.8	30.8	1.26544	.8	33.4	1.29462
.9	28.2	1.23777	.9	30.8	1.26601	.9	33.45	1.29521
51.0	28.2	1.23832	56.0	30.9	1.26658	61.0	33.5	1.29581
.1	28.3	1.23888	.1	30.9	1.26716	.1	33.6	1.29640
.2	28.35	1.23943	.2	31.0	1.26773	.2	33.6	1.29700
.3	28.4	1.23999	.3	31.05	1.26831	.3	33.7	1.29759
.4	28.5	1.24055	.4	31.1	1.26889	.4	33.7	1.29819
.5	28.5	1.24111	.5	31.2	1.26946	.5	33.8	1.29878
.6	28.6	1.24166	.6	31.2	1.27004	.6	33.8	1.29938
.7	28.6	1.24222	.7	31.3	1.27062	.7	33.9	1.29998
.8	28.7	1.24278	.8	31.3	1.27120	.8	33.9	1.30057
.9	28.7	1.24334	.9	31.4	1.27177	.9	34.0	1.30117
52.0	28.8	1.24390	57.0	31.4	1.27235	62.0	34.0	1.30177
.1	28.8	1.24446	.1	31.5	1.27293	.1	34.1	1.30237
.2	28.9	1.24502	.2	31.5	1.27351	.2	34.1	1.30297
.3	28.9	1.24558	.3	31.6	1.27409	.3	34.2	1.30356
.4	29.0	1.24614	.4	31.6	1.27464	.4	34.2	1.30416
.5	29.0	1.24670	.5	31.7	1.27525	.5	34.3	1.30476
.6	29.1	1.24726	.6	31.7	1.27583	.6	34.3	1.30536
.7	29.15	1.24782	.7	31.8	1.27641	.7	34.4	1.30596
.8	29.2	1.24839	.8	31.8	1.27699	.8	34.4	1.30657
.9	29.2	1.24895	.9	31.9	1.27758	.9	34.5	1.30717

TABLE 29—Continued

SHOWING A COMPARISON OF THE DEGREES BRIX AND BAUME, ETC.

Degree Brix (Per Cent Sugar)	Degree Baumé (Cor- rected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Cor- rected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Cor- rected)	Specific Gravity
63.0	34.5	1.30777	68.0	37.1	1.33836	73.0	39.6	1.36995
.1	34.6	1.30837	.1	37.1	1.33899	.1	39.7	1.37059
.2	34.6	1.30897	.2	37.2	1.33961	.2	39.7	1.37124
.3	34.7	1.30958	.3	37.3	1.34023	.3	39.8	1.37188
.4	34.7	1.31018	.4	37.3	1.34085	.4	39.8	1.37252
.4	34.8	1.31078	.5	37.4	1.34148	.5	39.9	1.37317
.6	34.85	1.31139	.6	37.4	1.34210	.6	39.9	1.37381
.7	34.9	1.31199	.7	37.5	1.34273	.7	40.0	1.37446
.8	34.95	1.31260	.8	37.5	1.34335	.8	40.0	1.37510
.9	35.0	1.31320	.9	37.6	1.34398	.9	40.1	1.37575
64.0	35.1	1.31381	69.0	37.6	1.34460	74.0	40.1	1.37639
.1	35.1	1.31442	.1	37.7	1.34523	.1	40.2	1.37704
.2	35.2	1.31502	.2	37.7	1.34585	.2	40.2	1.37768
.3	35.2	1.31563	.3	37.8	1.34648	.3	40.3	1.37833
.4	35.3	1.31624	.4	37.8	1.34711	.4	40.3	1.37898
.5	35.3	1.31684	.5	37.9	1.34774	.5	40.4	1.37962
.6	35.4	1.31745	.6	37.9	1.34836	.6	40.4	1.38027
.7	35.4	1.31806	.7	38.0	1.34899	.7	40.5	1.38092
.8	35.5	1.31867	.8	38.0	1.34962	.8	40.5	1.38157
.9	35.5	1.31928	.9	38.1	1.35025	.9	40.6	1.38222
65.0	35.6	1.31989	70.0	38.1	1.35088	75.0	40.6	1.38287
.1	35.6	1.32050	.1	38.2	1.35151	.1	40.7	1.38352
.2	35.7	1.32111	.2	38.2	1.35214	.2	40.7	1.38417
.3	35.7	1.32172	.3	38.3	1.35277	.3	40.8	1.38482
.4	35.8	1.32233	.4	38.3	1.35340	.4	40.8	1.38547
.5	35.8	1.32294	.5	38.4	1.35403	.5	40.9	1.38612
.6	35.9	1.32355	.6	38.4	1.35466	.6	40.9	1.38677
.7	35.9	1.32417	.7	38.5	1.35530	.7	41.0	1.38743
.8	36.0	1.32478	.8	38.5	1.35593	.8	41.0	1.38808
.9	36.0	1.32539	.9	38.6	1.35656	.9	41.1	1.38873
66.0	36.1	1.32601	71.0	38.6	1.35720	76.0	41.1	1.38939
.1	36.1	1.32662	.1	38.7	1.35783	.1	41.2	1.39004
.2	36.2	1.32724	.2	38.7	1.35847	.2	41.2	1.39070
.3	36.2	1.32785	.3	38.8	1.35910	.3	41.3	1.39135
.4	36.3	1.32847	.4	38.8	1.35974	.4	41.3	1.39201
.5	36.3	1.32908	.5	38.9	1.36037	.5	41.4	1.39266
.6	36.4	1.32970	.6	38.9	1.36101	.6	41.4	1.39332
.7	36.4	1.33031	.7	39.0	1.36164	.7	41.5	1.39397
.8	36.5	1.33093	.8	39.0	1.36228	.8	41.5	1.39463
.9	36.5	1.33155	.9	39.1	1.36292	.9	41.6	1.39529
67.0	36.6	1.33217	72.0	39.1	1.36355	77.0	41.6	1.39595
.1	36.6	1.33278	.1	39.2	1.36419	.1	41.7	1.39660
.2	36.7	1.33340	.2	39.2	1.36483	.2	41.7	1.39726
.3	36.75	1.33402	.3	39.3	1.36547	.3	41.8	1.39792
.4	36.8	1.33464	.4	39.3	1.36611	.4	41.8	1.39858
.5	36.85	1.33526	.5	39.4	1.36675	.5	41.9	1.39924
.6	36.9	1.33588	.6	39.4	1.36739	.6	41.9	1.39990
.7	36.95	1.33650	.7	39.5	1.36803	.7	42.0	1.40056
.8	37.0	1.33712	.8	39.5	1.36867	.8	42.0	1.40122
.9	37.0	1.33774	.9	39.6	1.36931	.9	42.1	1.40188

TABLE 29—Continued

SHOWING A COMPARISON OF THE DEGREES BRIX AND BAUMÉ, ETC.

Degree Brix (Per Cent Sugar)	Degree Baumé (Cor- rected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Cor- rected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Cor- rected)	Specific Gravity
78.0	42.1	1.40254	83.0	44.6	1.43614	88.0	47.0	1.47074
.1	42.2	1.40321	.1	44.6	1.43682	.1	47.0	1.47145
.2	42.2	1.40387	.2	44.7	1.43750	.2	47.1	1.47215
.3	42.3	1.40453	.3	44.7	1.43819	.3	47.1	1.47285
.4	42.3	1.40520	.4	44.8	1.43887	.4	47.2	1.47356
.5	42.4	1.40586	.5	44.8	1.43955	.5	47.2	1.47426
.6	42.4	1.40652	.6	44.9	1.44024	.6	47.3	1.47496
.7	42.5	1.40719	.7	44.9	1.44092	.7	47.3	1.47567
.8	42.5	1.40785	.8	45.0	1.44161	.8	47.4	1.47637
.9	42.6	1.40852	.9	45.0	1.44229	.9	47.4	1.47708
79.0	42.6	1.40918	84.0	45.1	1.44298	89.0	47.45	1.47778
.1	42.7	1.40985	.1	45.1	1.44367	.1	47.5	1.47849
.2	42.7	1.41052	.2	45.15	1.44435	.2	47.55	1.47920
.3	42.8	1.41118	.3	45.2	1.44504	.3	47.6	1.47991
.4	42.8	1.41185	.4	45.25	1.44573	.4	47.6	1.48061
.5	42.9	1.41252	.5	45.3	1.44641	.5	47.7	1.48132
.6	42.9	1.41318	.6	45.35	1.44710	.6	47.7	1.48203
.7	43.0	1.41385	.7	45.4	1.44779	.7	47.8	1.48274
.8	43.0	1.41452	.8	45.4	1.44848	.8	47.8	1.48345
.9	43.1	1.41519	.9	45.5	1.44917	.9	47.9	1.48416
80.0	43.1	1.41586	85.0	45.5	1.44986	90.0	47.9	1.48486
.1	43.2	1.41653	.1	45.6	1.45055	.1	48.0	1.48558
.2	43.2	1.41720	.2	45.6	1.45124	.2	48.0	1.48629
.3	43.2	1.41787	.3	45.7	1.45193	.3	48.1	1.48700
.4	43.3	1.41854	.4	45.7	1.45262	.4	48.1	1.48771
.5	43.3	1.41921	.5	45.8	1.45331	.5	48.2	1.48842
.6	43.4	1.41989	.6	45.8	1.45401	.6	48.2	1.48913
.7	43.45	1.42056	.7	45.9	1.45470	.7	48.3	1.48985
.8	43.5	1.42123	.8	45.9	1.45539	.8	48.3	1.49056
.9	43.55	1.42190	.9	46.0	1.45609	.9	48.35	1.49127
81.0	43.6	1.42258	86.0	46.0	1.45678	91.0	48.4	1.49199
.1	43.65	1.42325	.1	46.1	1.45748	.1	48.45	1.49270
.2	43.7	1.42393	.2	46.1	1.45817	.2	48.5	1.49342
.3	43.7	1.42460	.3	46.2	1.45887	.3	48.5	1.49413
.4	43.8	1.42528	.4	46.2	1.45956	.4	48.6	1.49485
.5	43.8	1.42595	.5	46.3	1.46026	.5	48.6	1.49556
.6	43.9	1.42663	.6	46.3	1.46095	.6	48.7	1.49628
.7	43.9	1.42731	.7	46.35	1.46165	.7	48.7	1.49700
.8	44.0	1.42798	.8	46.4	1.46235	.8	48.8	1.49771
.9	44.0	1.42866	.9	46.45	1.46304	.9	48.8	1.49843
82.0	44.1	1.42934	87.0	46.5	1.46374	92.0	48.9	1.49915
.1	44.1	1.43002	.1	46.55	1.46444	.1	48.9	1.49987
.2	44.2	1.43070	.2	46.6	1.46514	.2	49.0	1.50058
.3	44.2	1.43137	.3	46.65	1.46584	.3	49.0	1.50130
.4	44.3	1.43205	.4	46.7	1.46654	.4	49.05	1.50202
.5	44.3	1.43273	.5	46.7	1.46724	.5	49.1	1.50274
.6	44.4	1.43341	.6	46.8	1.46794	.6	49.15	1.50346
.7	44.4	1.43409	.7	46.8	1.46864	.7	49.2	1.50419
.8	44.5	1.43478	.8	46.9	1.46934	.8	49.2	1.50491
.9	44.5	1.43546	.9	46.9	1.47004	.9	49.3	1.50563

TABLE 29—Continued

SHOWING A COMPARISON OF THE DEGREES BRIX AND BAUME, ETC.

Degree Brix (Per Cent Sugar)	Degree Baumé (Corrected)	Specific Gravity	Degree Brix (Per Cent Sugar)	Degree Baumé (Corrected)	Specific Gravity
93.0	49.3	1.50635	94.0	49.8	1.51359
.1	49.4	1.50707	.1	49.85	1.51431
.2	49.4	1.50779	.2	49.9	1.51504
.3	49.5	1.50852	.3	49.9	1.51577
.4	49.5	1.50924	.4	50.0	1.51649
.5	49.6	1.50996	.5	50.0	1.51722
.6	49.6	1.51069	.6	50.1	1.51795
.7	49.7	1.51141	.7	50.1	1.51868
.8	49.7	1.51214	.8	50.2	1.51941
.9	49.8	1.51286	.9	50.2	1.52014
			95.0	50.3	1.52087

TABLE 30

FOR THE CORRECTION OF READINGS ON THE BRIX SCALE FOR VARIATIONS IN TEMPERATURE FROM THE STANDARD, 17½° C. (63½° F.)—(GERLACH)

Temp. ° C.	Temp. ° F.	Approximate Degree Brix and Correction													
		0	5	10	15	20	25	30	35	40	50	60	70	75	
0	32	.27	.30	.41	.52	.62	.72	.82	.92	.98	1.11	1.22	1.25	1.29	
5	41	.23	.30	.37	.44	.52	.59	.65	.72	.75	.80	.88	.91	.94	
10	50	.20	.26	.29	.33	.36	.39	.42	.45	.48	.50	.54	.58	.61	
11	51.8	.18	.23	.26	.28	.31	.34	.36	.39	.41	.43	.47	.50	.53	
12	53.6	.16	.20	.22	.24	.26	.29	.31	.33	.34	.36	.40	.42	.46	
13	55.4	.14	.18	.19	.21	.22	.24	.26	.27	.28	.29	.33	.35	.39	
14	57.2	.12	.15	.16	.17	.18	.19	.21	.22	.22	.23	.26	.28	.32	
15	59	.09	.11	.12	.14	.14	.15	.16	.17	.17	.17	.19	.21	.25	
16	60.8	.06	.07	.08	.09	.10	.10	.11	.12	.12	.12	.14	.16	.18	
17	62.6	.02	.02	.03	.03	.03	.04	.04	.04	.04	.04	.05	.05	.06	

Add the correction to readings above 17½° C. (63½° F.) and subtract the correction from those below this temperature

18	64.4	.02	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.03	.02
19	66.2	.06	.08	.08	.09	.09	.10	.10	.10	.10	.10	.10	.08	.06
20	68	.11	.14	.15	.17	.17	.18	.18	.18	.19	.19	.18	.15	.11
21	69.8	.16	.20	.22	.24	.24	.25	.25	.25	.26	.26	.25	.22	.18
22	71.6	.21	.26	.29	.31	.31	.32	.32	.32	.33	.34	.32	.29	.25
23	73.4	.27	.32	.35	.37	.38	.39	.39	.39	.40	.42	.39	.36	.33
24	75.2	.32	.38	.41	.43	.44	.46	.46	.47	.47	.50	.46	.43	.40
25	77	.37	.44	.47	.49	.51	.53	.54	.55	.55	.58	.54	.51	.48
26	78.8	.43	.50	.54	.56	.58	.60	.61	.62	.62	.66	.62	.58	.55
27	80.6	.49	.57	.61	.63	.65	.68	.68	.69	.70	.74	.70	.65	.62
28	82.4	.56	.64	.68	.70	.72	.76	.76	.78	.78	.82	.78	.72	.70
29	84.2	.63	.71	.75	.78	.79	.84	.84	.86	.86	.90	.86	.80	.78
30	86	.70	.78	.82	.87	.87	.92	.92	.94	.94	.98	.94	.88	.86
35	95	1.10	1.17	1.22	1.24	1.30	1.32	1.33	1.35	1.36	1.39	1.34	1.27	1.25
40	104	1.50	1.61	1.67	1.71	1.73	1.79	1.79	1.80	1.82	1.83	1.78	1.69	1.65
50	122	....	2.65	2.71	2.74	2.78	2.80	2.80	2.80	2.80	2.79	2.70	2.56	2.51
60	140	....	3.87	3.88	3.88	3.88	3.88	3.88	3.88	3.90	3.82	3.70	3.43	3.41
70	158	....	....	5.18	5.20	5.14	5.13	5.10	5.08	5.06	4.90	4.72	4.47	4.35
80	176	....	....	6.62	6.59	6.54	6.46	6.38	6.30	6.26	6.06	5.82	5.50	5.33

TABLE 31

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS<sup>1</sup>  
AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
0.0	0.99823	1.00000	0.00	5.0	1.01785	1.01965	2.79
.1	0.99862	1.00039	0.06	.1	1.01825	1.02005	2.85
.2	0.99901	1.00078	0.11	.2	1.01865	1.02045	2.91
.3	0.99940	1.00117	0.17	.3	1.01905	1.02085	2.96
.4	0.99979	1.00155	0.22	.4	1.01945	1.02125	3.02
0.5	1.00017	1.00194	0.28	5.5	1.01985	1.02165	3.07
.6	1.00056	1.00233	0.34	.6	1.02025	1.02206	3.13
.7	1.00095	1.00272	0.39	.7	1.02065	1.02246	3.18
.8	1.00134	1.00311	0.45	.8	1.02105	1.02286	3.24
.9	1.00173	1.00350	0.51	.9	1.02145	1.02321	3.30
1.0	1.00212	1.00389	0.56	6.0	1.02186	1.02366	3.35
.1	1.00251	1.00428	0.62	.1	1.02226	1.02407	3.41
.2	1.00290	1.00467	0.67	.2	1.02266	1.02447	3.46
.3	1.00329	1.00506	0.73	.3	1.02306	1.02487	3.52
.4	1.00368	1.00545	0.79	.4	1.02346	1.02527	3.57
1.5	1.00406	1.00584	0.84	6.5	1.02387	1.02568	3.63
.6	1.00445	1.00623	0.90	.6	1.02427	1.02608	3.69
.7	1.00484	1.00662	0.95	.7	1.02467	1.02648	3.74
.8	1.00523	1.00701	1.01	.8	1.02508	1.02689	3.80
.9	1.00562	1.00740	1.07	.9	1.02548	1.02729	3.85
2.0	1.00602	1.00779	1.12	7.0	1.02588	1.02770	3.91
.1	1.00641	1.00818	1.18	.1	1.02629	1.02810	3.96
.2	1.00680	1.00858	1.23	.2	1.02669	1.02851	4.02
.3	1.00719	1.00897	1.29	.3	1.02710	1.02892	4.08
.4	1.00758	1.00936	1.34	.4	1.02750	1.02932	4.13
2.5	1.00797	1.00976	1.40	7.5	1.02791	1.02973	4.19
.6	1.00836	1.01015	1.46	.6	1.02832	1.03013	4.24
.7	1.00876	1.01054	1.51	.7	1.02872	1.03054	4.30
.8	1.00915	1.01093	1.57	.8	1.02913	1.03095	4.35
.9	1.00954	1.01133	1.62	.9	1.02954	1.03136	4.41
3.0	1.00993	1.01172	1.68	8.0	1.02994	1.03176	4.46
.1	1.01033	1.01211	1.74	.1	1.03035	1.03217	4.52
.2	1.01072	1.01251	1.79	.2	1.03076	1.03258	4.58
.3	1.01112	1.01290	1.85	.3	1.03116	1.03299	4.63
.4	1.01151	1.01330	1.90	.4	1.03157	1.03340	4.69
3.5	1.01190	1.01369	1.96	8.5	1.03198	1.03381	4.74
.6	1.01230	1.01409	2.02	.6	1.03239	1.03422	4.80
.7	1.01269	1.01448	2.07	.7	1.03280	1.03463	4.85
.8	1.01309	1.01488	2.13	.8	1.03321	1.03504	4.91
.9	1.01348	1.01528	2.18	.9	1.03362	1.03545	4.96
4.0	1.01388	1.01567	2.24	9.0	1.03403	1.03586	5.02
.1	1.01428	1.01607	2.29	.1	1.03444	1.03627	5.07
.2	1.01467	1.01647	2.35	.2	1.03485	1.03668	5.13
.3	1.01507	1.01687	2.40	.3	1.03526	1.03709	5.19
.4	1.01547	1.01726	2.46	.4	1.03567	1.03750	5.24
4.5	1.01586	1.01766	2.52	9.5	1.03608	1.03792	5.30
.6	1.01626	1.01806	2.57	.6	1.03649	1.03833	5.35
.7	1.01666	1.01846	2.63	.7	1.03691	1.03874	5.41
.8	1.01706	1.01886	2.68	.8	1.03732	1.03915	5.46
.9	1.01746	1.01926	2.74	.9	1.03773	1.03957	5.52

<sup>1</sup>From Bureau Standards Circular 44 (1918).

TABLE 31—Continued

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS  
AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
10.0	1.03814	1.03998	5.57	15.0	1.05916	1.06104	8.34
.1	1.03856	1.04039	5.63	.1	1.05959	1.06147	8.40
.2	1.03897	1.04081	5.68	.2	1.06002	1.06190	8.45
.3	1.03938	1.04122	5.74	.3	1.06045	1.06233	8.51
.4	1.03980	1.04164	5.80	.4	1.06088	1.06276	8.56
10.5	1.04021	1.04205	5.85	15.5	1.06131	1.06319	8.62
.6	1.04063	1.04247	5.91	.6	1.06174	1.06362	8.67
.7	1.04104	1.04288	5.96	.7	1.06217	1.06405	8.73
.8	1.04146	1.04330	6.02	.8	1.06260	1.06448	8.78
.9	1.04187	1.04371	6.07	.9	1.06303	1.06491	8.84
11.0	1.04229	1.04413	6.13	16.0	1.06346	1.06534	8.89
.1	1.04270	1.04455	6.18	.1	1.06389	1.06577	8.95
.2	1.04312	1.04497	6.24	.2	1.06432	1.06621	9.00
.3	1.04354	1.04538	6.30	.3	1.06476	1.06664	9.06
.4	1.04395	1.04580	6.35	.4	1.06519	1.06707	9.11
11.5	1.04437	1.04622	6.41	16.5	1.06562	1.06751	9.17
.6	1.04479	1.04664	6.46	.6	1.06605	1.06794	9.22
.7	1.04521	1.04706	6.52	.7	1.06649	1.06837	9.28
.8	1.04562	1.04747	6.57	.8	1.06692	1.06881	9.33
.9	1.04604	1.04789	6.63	.9	1.06736	1.06924	9.39
12.0	1.04646	1.04831	6.68	17.0	1.06779	1.06968	9.45
.1	1.04688	1.04873	6.74	.1	1.06822	1.07011	9.50
.2	1.04730	1.04915	6.79	.2	1.06866	1.07055	9.56
.3	1.04772	1.04957	6.85	.3	1.06909	1.07098	9.61
.4	1.04814	1.04999	6.90	.4	1.06953	1.07142	9.67
12.5	1.04856	1.05041	6.96	17.5	1.06996	1.07186	9.72
.6	1.04898	1.05084	7.02	.6	1.07040	1.07229	9.78
.7	1.04940	1.05126	7.07	.7	1.07084	1.07273	9.83
.8	1.04982	1.05168	7.13	.8	1.07127	1.07317	9.89
.9	1.05024	1.05210	7.18	.9	1.07171	1.07361	9.94
13.0	1.05066	1.05252	7.24	18.0	1.07215	1.07404	10.00
.1	1.05109	1.05295	7.29	.1	1.07258	1.07448	10.05
.2	1.05151	1.05337	7.35	.2	1.07302	1.07492	10.11
.3	1.05193	1.05379	7.40	.3	1.07346	1.07536	10.16
.4	1.05236	1.05422	7.46	.4	1.07390	1.07580	10.22
13.5	1.05278	1.05464	7.51	18.5	1.07434	1.07624	10.27
.6	1.05320	1.05506	7.57	.6	1.07478	1.07668	10.33
.7	1.05363	1.05549	7.62	.7	1.07522	1.07712	10.38
.8	1.05405	1.05591	7.68	.8	1.07566	1.07756	10.44
.9	1.05448	1.05634	7.73	.9	1.07610	1.07800	10.49
14.0	1.05490	1.05677	7.79	19.0	1.07654	1.07844	10.55
.1	1.05532	1.05719	7.84	.1	1.07698	1.07888	10.60
.2	1.05575	1.05762	7.90	.2	1.07742	1.07932	10.66
.3	1.05618	1.05804	7.96	.3	1.07786	1.07977	10.71
.4	1.05660	1.05847	8.01	.4	1.07830	1.08021	10.77
14.5	1.05703	1.05890	8.07	19.5	1.07874	1.08065	10.82
.6	1.05746	1.05933	8.12	.6	1.07919	1.08110	10.88
.7	1.05788	1.05975	8.18	.7	1.07963	1.08154	10.93
.8	1.05831	1.06018	8.23	.8	1.08007	1.08198	10.99
.9	1.05874	1.06061	8.29	.9	1.08052	1.08243	11.04

TABLE 31—Continued

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS  
AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé at (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé at (Modulus 145)
20.0	1.08096	1.08287	11.10	25.0	1.10356	1.10551	13.84
.1	1.08140	1.08332	11.15	.1	1.10402	1.10597	13.89
.2	1.08185	1.08376	11.21	.2	1.10448	1.10643	13.95
.3	1.08229	1.08421	11.26	.3	1.10494	1.10689	14.00
.4	1.08274	1.08465	11.32	.4	1.10540	1.10736	14.06
20.5	1.08318	1.08510	11.37	25.5	1.10586	1.10782	14.11
.6	1.08363	1.08554	11.43	.6	1.10632	1.10828	14.17
.7	1.08407	1.08599	11.48	.7	1.10679	1.10874	14.22
.8	1.08452	1.08644	11.54	.8	1.10725	1.10921	14.28
.9	1.08497	1.08689	11.59	.9	1.10771	1.10967	14.33
21.0	1.08541	1.08733	11.65	26.0	1.10818	1.11014	14.39
.1	1.08586	1.08778	11.70	.1	1.10864	1.11060	14.44
.2	1.08631	1.08823	11.76	.2	1.10910	1.11106	14.49
.3	1.08676	1.08868	11.81	.3	1.10957	1.11153	14.55
.4	1.08720	1.08913	11.87	.4	1.11003	1.11200	14.60
21.5	1.08765	1.08958	11.92	25.5	1.11050	1.11246	14.66
.6	1.08810	1.09003	11.98	.6	1.11096	1.11293	14.71
.7	1.08855	1.09048	12.03	.7	1.11143	1.11339	14.77
.8	1.08900	1.09093	12.09	.8	1.11190	1.11386	14.82
.9	1.08945	1.09138	12.14	.9	1.11236	1.11433	14.88
22.0	1.08990	1.09183	12.20	27.0	1.11283	1.11480	14.93
.1	1.09035	1.09228	12.25	.1	1.11330	1.11526	14.99
.2	1.09080	1.09273	12.31	.2	1.11376	1.11573	15.04
.3	1.09125	1.09318	12.36	.3	1.11423	1.11620	15.09
.4	1.09170	1.09364	12.42	.4	1.11470	1.11667	15.15
22.5	1.09216	1.09409	12.47	27.5	1.11517	1.11714	15.20
.6	1.09261	1.09454	12.52	.6	1.11564	1.11761	15.26
.7	1.09306	1.09499	12.58	.7	1.11610	1.11808	15.31
.8	1.09351	1.09545	12.63	.8	1.11657	1.11855	15.37
.9	1.09397	1.09590	12.69	.9	1.11704	1.11902	15.42
23.0	1.09442	1.09636	12.74	28.0	1.11751	1.11949	15.48
.1	1.09487	1.09681	12.80	.1	1.11798	1.11996	15.53
.2	1.09533	1.09727	12.85	.2	1.11845	1.12043	15.59
.3	1.09578	1.09772	12.91	.3	1.11892	1.12090	15.64
.4	1.09624	1.09818	12.96	.4	1.11940	1.12138	15.69
23.5	1.09669	1.09863	13.02	28.5	1.11987	1.12185	15.75
.6	1.09715	1.09909	13.07	.6	1.12034	1.12232	15.80
.7	1.09760	1.09954	13.13	.7	1.12081	1.12280	15.86
.8	1.09806	1.10000	13.18	.8	1.12128	1.12327	15.91
.9	1.09851	1.10046	13.24	.9	1.12176	1.12374	15.97
24.0	1.09897	1.10092	13.29	29.0	1.12223	1.12422	16.02
.1	1.09943	1.10137	13.35	.1	1.12270	1.12469	16.08
.2	1.09989	1.10183	13.40	.2	1.12318	1.12517	16.13
.3	1.10034	1.10229	13.46	.3	1.12365	1.12564	16.18
.4	1.10080	1.10275	13.51	.4	1.12413	1.12612	16.24
24.5	1.10126	1.10321	13.57	29.5	1.12460	1.12659	16.29
.6	1.10172	1.10367	13.62	.6	1.12508	1.12707	16.35
.7	1.10218	1.10413	13.67	.7	1.12556	1.12755	16.40
.8	1.10264	1.10459	13.73	.8	1.12603	1.12802	16.46
.9	1.10310	1.10505	13.78	.9	1.12651	1.12850	16.51

TABLE 31—Continued

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS  
AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
30.0	1.12698	1.12898	16.57	35.0	1.15128	1.15331	19.28
.1	1.12746	1.12946	16.62	.1	1.15177	1.15381	19.33
.2	1.12794	1.12993	16.67	.2	1.15226	1.15430	19.38
.3	1.12842	1.13041	16.73	.3	1.15276	1.15480	19.44
.4	1.12890	1.13089	16.78	.4	1.15326	1.15530	19.49
30.5	1.12937	1.13137	16.84	35.5	1.15375	1.15579	19.55
.6	1.12985	1.13185	16.89	.6	1.15425	1.15629	19.60
.7	1.13033	1.13233	16.95	.7	1.15475	1.15679	19.65
.8	1.13081	1.13281	17.00	.8	1.15524	1.15729	19.71
.9	1.13129	1.13329	17.05	.9	1.15574	1.15778	19.76
31.0	1.13177	1.13378	17.11	36.0	1.15624	1.15828	19.81
.1	1.13225	1.13426	17.16	.1	1.15674	1.15878	19.87
.2	1.13274	1.13474	17.22	.2	1.15724	1.15928	19.92
.3	1.13322	1.13522	17.27	.3	1.15773	1.15978	19.98
.4	1.13370	1.13570	17.33	.4	1.15823	1.16028	20.03
31.5	1.13418	1.13619	17.38	36.5	1.15873	1.16078	20.08
.6	1.13466	1.13667	17.43	.6	1.15923	1.16128	20.14
.7	1.13515	1.13715	17.49	.7	1.15973	1.16178	20.19
.8	1.13563	1.13764	17.54	.8	1.16023	1.16228	20.25
.9	1.13611	1.13812	17.60	.9	1.16073	1.16279	20.30
32.0	1.13660	1.13861	17.65	37.0	1.16124	1.16329	20.35
.1	1.13708	1.13909	17.70	.1	1.16174	1.16379	20.41
.2	1.13756	1.13958	17.76	.2	1.16224	1.16430	20.46
.3	1.13805	1.14006	17.81	.3	1.16274	1.16480	20.52
.4	1.13853	1.14055	17.87	.4	1.16324	1.16530	20.57
32.5	1.13902	1.14103	17.92	37.5	1.16375	1.16581	20.62
.6	1.13951	1.14152	17.98	.6	1.16425	1.16631	20.68
.7	1.13999	1.14201	18.03	.7	1.16476	1.16682	20.73
.8	1.14048	1.14250	18.08	.8	1.16526	1.16732	20.78
.9	1.14097	1.14298	18.14	.9	1.16576	1.16783	20.84
33.0	1.14145	1.14347	18.19	38.0	1.16627	1.16833	20.89
.1	1.14194	1.14396	18.25	.1	1.16678	1.16884	20.94
.2	1.14243	1.14445	18.30	.2	1.16728	1.16934	21.00
.3	1.14292	1.14494	18.36	.3	1.16779	1.16985	21.05
.4	1.14340	1.14543	18.41	.4	1.16829	1.17036	21.11
33.5	1.14389	1.14592	18.46	38.5	1.16880	1.17087	21.16
.6	1.14438	1.14641	18.52	.6	1.16931	1.17138	21.21
.7	1.14487	1.14690	18.57	.7	1.16982	1.17188	21.27
.8	1.14536	1.14739	18.63	.8	1.17032	1.17239	21.32
.9	1.14585	1.14788	18.68	.9	1.17083	1.17290	21.38
34.0	1.14634	1.14837	18.73	39.0	1.17134	1.17341	21.43
.1	1.14684	1.14886	18.79	.1	1.17185	1.17392	21.48
.2	1.14733	1.14936	18.84	.2	1.17236	1.17443	21.54
.3	1.14782	1.14985	18.90	.3	1.17287	1.17494	21.59
.4	1.14831	1.15034	18.95	.4	1.17338	1.17545	21.64
34.5	1.14880	1.15084	19.00	39.5	1.17389	1.17596	21.70
.6	1.14930	1.15133	19.06	.6	1.17440	1.17648	21.75
.7	1.14979	1.15183	19.11	.7	1.17491	1.17699	21.80
.8	1.15028	1.15232	19.17	.8	1.17542	1.17750	21.86
.9	1.15078	1.15282	19.22	.9	1.17594	1.17802	21.91



TABLE 31—Continued

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS  
AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé at (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé at (Modulus 145)
40.0	1.17645	1.17853	21.97	45.0	1.20254	1.20467	24.63
.1	1.17696	1.17904	22.02	.1	1.20307	1.20520	24.69
.2	1.17747	1.17956	22.07	.2	1.20360	1.20573	24.74
.3	1.17799	1.18007	22.13	.3	1.20414	1.20627	24.79
.4	1.17850	1.18058	22.18	.4	1.20467	1.20680	24.85
40.5	1.17901	1.18110	22.23	45.5	1.20520	1.20733	24.90
.6	1.17953	1.18162	22.29	.6	1.20573	1.20787	24.95
.7	1.18004	1.18213	22.34	.7	1.20627	1.20840	25.01
.8	1.18056	1.18265	22.39	.8	1.20680	1.20894	25.06
.9	1.18108	1.18316	22.45	.9	1.20734	1.20947	25.11
41.0	1.18159	1.18368	22.50	46.0	1.20787	1.21001	25.17
.1	1.18211	1.18420	22.55	.1	1.20840	1.21054	25.22
.2	1.18262	1.18472	22.61	.2	1.20894	1.21108	25.27
.3	1.18314	1.18524	22.66	.3	1.20948	1.21162	25.32
.4	1.18366	1.18575	22.72	.4	1.21001	1.21215	25.38
41.5	1.18418	1.18627	22.77	46.5	1.21055	1.21269	25.43
.6	1.18470	1.18679	22.82	.6	1.21109	1.21323	25.48
.7	1.18522	1.18731	22.88	.7	1.21162	1.21377	25.54
.8	1.18573	1.18783	22.93	.8	1.21216	1.21431	25.59
.9	1.18625	1.18835	22.98	.9	1.21270	1.21484	25.64
42.0	1.18677	1.18887	23.04	47.0	1.21324	1.21538	25.70
.1	1.18729	1.18939	23.09	.1	1.21378	1.21592	25.75
.2	1.18781	1.18992	23.14	.2	1.21432	1.21646	25.80
.3	1.18834	1.19044	23.20	.3	1.21486	1.21700	25.86
.4	1.18886	1.19096	23.25	.4	1.21540	1.21755	25.91
42.5	1.18938	1.19148	23.30	47.5	1.21594	1.21809	25.96
.6	1.18990	1.19201	23.36	.6	1.21648	1.21863	26.01
.7	1.19042	1.19253	23.41	.7	1.21702	1.21917	26.07
.8	1.19095	1.19305	23.46	.8	1.21756	1.21971	26.12
.9	1.19147	1.19358	23.52	.9	1.21810	1.22026	26.17
43.0	1.19199	1.19410	23.57	48.0	1.21864	1.22080	26.23
.1	1.19252	1.19463	23.62	.1	1.21918	1.22134	26.28
.2	1.19304	1.19515	23.68	.2	1.21973	1.22189	26.33
.3	1.19356	1.19568	23.73	.3	1.22027	1.22243	26.38
.4	1.19409	1.19620	23.78	.4	1.22082	1.22298	26.44
43.5	1.19462	1.19673	23.84	48.5	1.22136	1.22352	26.49
.6	1.19514	1.19726	23.89	.6	1.22190	1.22406	26.54
.7	1.19567	1.19778	23.94	.7	1.22245	1.22461	26.59
.8	1.19619	1.19831	24.00	.8	1.22300	1.22516	26.65
.9	1.19672	1.19884	24.05	.9	1.22354	1.22570	26.70
44.0	1.19725	1.19936	24.10	49.0	1.22409	1.22625	26.75
.1	1.19778	1.19989	24.16	.1	1.22463	1.22680	26.81
.2	1.19830	1.20042	24.21	.2	1.22518	1.22735	26.86
.3	1.19883	1.20095	24.26	.3	1.22573	1.22789	26.91
.4	1.19936	1.20148	24.32	.4	1.22627	1.22844	26.96
44.5	1.19989	1.20201	24.37	49.5	1.22682	1.22899	27.02
.6	1.20042	1.20254	24.42	.6	1.22737	1.22954	27.07
.7	1.20095	1.20307	24.48	.7	1.22792	1.23009	27.12
.8	1.20148	1.20360	24.53	.8	1.22847	1.23064	27.18
.9	1.20201	1.20414	24.58	.9	1.22902	1.23119	27.23

TABLE 31—Continued

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS  
AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4 C°.	Specific Gravity at 20°/20° C.	Degrees Baumé at (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4 C°.	Specific Gravity at 20°/20° C.	Degrees Baumé at (Modulus 145)
<b>70.0</b>	1.34717	1.34956	37.56	<b>75.0</b>	1.37897	1.38141	40.03
.1	1.34780	1.35019	37.61	.1	1.37962	1.38206	40.08
.2	1.34843	1.35081	37.66	.2	1.38026	1.38270	40.13
.3	1.34906	1.35144	37.71	.3	1.38091	1.38335	40.18
.4	1.34968	1.35207	37.76	.4	1.38156	1.38400	40.23
<b>70.5</b>	1.35031	1.35270	37.81	<b>75.5</b>	1.38220	1.38465	40.28
.6	1.35094	1.35333	37.86	.6	1.38285	1.38530	40.33
.7	1.35157	1.35396	37.91	.7	1.38350	1.38595	40.38
.8	1.35220	1.35459	37.96	.8	1.38415	1.38660	40.43
.9	1.35283	1.35522	38.01	.9	1.38480	1.38725	40.48
<b>71.0</b>	1.35346	1.35585	38.06	<b>76.0</b>	1.38545	1.38790	40.53
.1	1.35409	1.35648	38.11	.1	1.38610	1.38855	40.57
.2	1.35472	1.35711	38.16	.2	1.38675	1.38920	40.62
.3	1.35535	1.35775	38.21	.3	1.38740	1.38985	40.67
.4	1.35598	1.35838	38.26	.4	1.38805	1.39050	40.72
<b>71.5</b>	1.35661	1.35901	38.30	<b>76.5</b>	1.38870	1.39115	40.77
.6	1.35724	1.35964	38.35	.6	1.38935	1.39180	40.82
.7	1.35788	1.36028	38.40	.7	1.39000	1.39246	40.87
.8	1.35851	1.36091	38.45	.8	1.39065	1.39311	40.92
.9	1.35914	1.36155	38.50	.9	1.39130	1.39376	40.97
<b>72.0</b>	1.35978	1.36218	38.55	<b>77.0</b>	1.39196	1.39442	41.01
.1	1.36041	1.36282	38.60	.1	1.39261	1.39507	41.06
.2	1.36105	1.36346	38.65	.2	1.39326	1.39573	41.11
.3	1.36168	1.36409	38.70	.3	1.39392	1.39638	41.16
.4	1.36232	1.36473	38.75	.4	1.39457	1.39704	41.21
<b>72.5</b>	1.36295	1.36536	38.80	<b>77.5</b>	1.39523	1.39769	41.26
.6	1.36359	1.36600	38.85	.6	1.39588	1.39835	41.31
.7	1.36423	1.36664	38.90	.7	1.39654	1.39901	41.36
.8	1.36486	1.36728	38.95	.8	1.39719	1.39966	41.40
.9	1.36550	1.36792	39.00	.9	1.39785	1.40032	41.45
<b>73.0</b>	1.36614	1.36856	39.05	<b>78.0</b>	1.39850	1.40098	41.50
.1	1.36678	1.36919	39.10	.1	1.39916	1.40164	41.55
.2	1.36742	1.36983	39.15	.2	1.39982	1.40230	41.60
.3	1.36805	1.37047	39.20	.3	1.40048	1.40295	41.65
.4	1.36869	1.37111	39.25	.4	1.40113	1.40361	41.70
<b>73.5</b>	1.36933	1.37176	39.30	<b>78.5</b>	1.40179	1.40427	41.74
.6	1.36997	1.37240	39.35	.6	1.40245	1.40493	41.79
.7	1.37061	1.37304	39.39	.7	1.40311	1.40559	41.84
.8	1.37125	1.37368	39.44	.8	1.40377	1.40625	41.89
.9	1.37189	1.37432	39.49	.9	1.40443	1.40691	41.94
<b>74.0</b>	1.37254	1.37496	39.54	<b>79.0</b>	1.40509	1.40758	41.99
.1	1.37318	1.37561	39.59	.1	1.40575	1.40824	42.03
.2	1.37382	1.37625	39.64	.2	1.40641	1.40890	42.08
.3	1.37446	1.37689	39.69	.3	1.40707	1.40956	42.13
.4	1.37510	1.37754	39.74	.4	1.40774	1.41023	42.18
<b>74.5</b>	1.37575	1.37818	39.79	<b>79.5</b>	1.40840	1.41089	42.23
.6	1.37639	1.37883	39.84	.6	1.40906	1.41155	42.28
.7	1.37704	1.37947	39.89	.7	1.40972	1.41222	42.32
.8	1.37768	1.38012	39.94	.8	1.41039	1.41288	42.37
.9	1.37833	1.38076	39.99	.9	1.41105	1.41355	42.42

TABLE 31—Continued

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUMÉ OF SUGAR SOLUTIONS  
AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé at (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
80.0	1.41172	1.41421	42.47	85.0	1.44539	1.44794	44.86
.1	1.41238	1.41488	42.52	.1	1.44607	1.44863	44.91
.2	1.41304	1.41554	42.57	.2	1.44675	1.44931	44.95
.3	1.41371	1.41621	42.61	.3	1.44744	1.45000	45.00
.4	1.41437	1.41688	42.66	.4	1.44812	1.45068	45.05
80.5	1.41504	1.41754	42.71	85.5	1.44881	1.45137	45.09
.6	1.41571	1.41821	42.76	.6	1.44949	1.45205	45.14
.7	1.41637	1.41888	42.81	.7	1.45018	1.45274	45.19
.8	1.41704	1.41955	42.85	.8	1.45086	1.45343	45.24
.9	1.41771	1.42022	42.90	.9	1.45154	1.45411	45.28
81.0	1.41837	1.42088	42.95	86.0	1.45223	1.45480	45.33
.1	1.41904	1.42155	43.00	.1	1.45292	1.45549	45.38
.2	1.41971	1.42222	43.05	.2	1.45360	1.45618	45.42
.3	1.42038	1.42289	43.10	.3	1.45429	1.45686	45.47
.4	1.42105	1.42356	43.14	.4	1.45498	1.45755	45.52
81.5	1.42172	1.42423	43.19	86.5	1.45567	1.45824	45.57
.6	1.42239	1.42490	43.24	.6	1.45636	1.45893	45.61
.7	1.42306	1.42558	43.29	.7	1.45704	1.45962	45.66
.8	1.42373	1.42625	43.33	.8	1.45773	1.46031	45.71
.9	1.42440	1.42692	43.38	.9	1.45842	1.46100	45.75
82.0	1.42507	1.42759	43.43	87.0	1.45911	1.46170	45.80
.1	1.42574	1.42827	43.48	.1	1.45980	1.46239	45.85
.2	1.42642	1.42894	43.53	.2	1.46050	1.46308	45.89
.3	1.42709	1.42961	43.57	.3	1.46119	1.46377	45.94
.4	1.42776	1.43029	43.62	.4	1.46188	1.46446	45.99
82.5	1.42844	1.43096	43.67	87.5	1.46257	1.46516	46.03
.6	1.42911	1.43164	43.72	.6	1.46326	1.46585	46.08
.7	1.42978	1.43231	43.77	.7	1.46395	1.46654	46.13
.8	1.43046	1.43298	43.81	.8	1.46464	1.46724	46.17
.9	1.43113	1.43366	43.86	.9	1.46534	1.46793	46.22
83.0	1.43181	1.43434	43.91	88.0	1.46603	1.46862	46.27
.1	1.43248	1.43502	43.95	.1	1.46673	1.46932	46.31
.2	1.43316	1.43569	44.00	.2	1.46742	1.47002	46.36
.3	1.43384	1.43637	44.05	.3	1.46812	1.47071	46.41
.4	1.43451	1.43705	44.10	.4	1.46881	1.47141	46.45
83.5	1.43519	1.43773	44.15	88.5	1.46950	1.47210	46.50
.6	1.43587	1.43841	44.19	.6	1.47020	1.47280	46.55
.7	1.43654	1.43908	44.24	.7	1.47090	1.47350	46.59
.8	1.43722	1.43976	44.29	.8	1.47159	1.47420	46.64
.9	1.43790	1.44044	44.34	.9	1.47229	1.47489	46.69
84.0	1.43858	1.44112	44.38	89.0	1.47299	1.47559	46.73
.1	1.43926	1.44180	44.43	.1	1.47368	1.47629	46.78
.2	1.43994	1.44249	44.48	.2	1.47438	1.47699	46.83
.3	1.44062	1.44317	44.53	.3	1.47508	1.47769	46.87
.4	1.44130	1.44385	44.57	.4	1.47578	1.47839	46.92
84.5	1.44198	1.44453	44.62	89.5	1.47648	1.47909	46.97
.6	1.44266	1.44521	44.67	.6	1.47718	1.47979	47.01
.7	1.44334	1.44590	44.72	.7	1.47788	1.48049	47.06
.8	1.44402	1.44658	44.76	.8	1.47858	1.48119	47.11
.9	1.44470	1.44726	44.81	.9	1.47928	1.48189	47.15

TABLE 31—Continued

DEGREES BRIX, SPECIFIC GRAVITY, AND DEGREES BAUME OF SUGAR SOLUTIONS  
AT 20° C.

Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)	Degrees Brix or Per Cent Sucrose by Weight	Specific Gravity at 20°/4° C.	Specific Gravity at 20°/20° C.	Degrees Baumé (Modulus 145)
90.0	1.47998	1.48259	47.20	95.0	1.51546	1.51814	49.49
.1	1.48008	1.48330	47.24	.1	1.51617	1.51886	49.53
.2	1.48138	1.48400	47.29	.2	1.51689	1.51958	49.58
.3	1.48208	1.48470	47.34	.3	1.51761	1.52030	49.62
.4	1.48278	1.48540	47.38	.4	1.51833	1.52102	49.67
90.5	1.48348	1.48611	47.43	95.5	1.51905	1.52174	49.71
.6	1.48419	1.48681	47.48	.6	1.51977	1.52246	49.76
.7	1.48489	1.48752	47.52	.7	1.52049	1.52318	49.80
.8	1.48559	1.48822	47.57	.8	1.52121	1.52390	49.85
.9	1.48630	1.48893	47.61	.9	1.52193	1.52463	49.90
91.0	1.48700	1.48963	47.66	96.0	1.52266	1.52535	49.94
.1	1.48771	1.49034	47.71	.1	1.52338	1.52607	49.98
.2	1.48841	1.49104	47.75	.2	1.52410	1.52680	50.03
.3	1.48912	1.49175	47.80	.3	1.52482	1.52752	50.08
.4	1.48982	1.49246	47.84	.4	1.52555	1.52824	50.12
91.5	1.49053	1.49316	47.89	96.5	1.52627	1.52897	50.16
.6	1.49123	1.49387	47.94	.6	1.52699	1.52969	50.21
.7	1.49194	1.49458	47.98	.7	1.52772	1.53042	50.25
.8	1.49265	1.49529	48.03	.8	1.52844	1.53114	50.30
.9	1.49336	1.49600	48.08	.9	1.52917	1.53187	50.34
92.0	1.49406	1.49671	48.12	97.0	1.52989	1.53260	50.39
.1	1.49477	1.49741	48.17	.1	1.53062	1.53332	50.43
.2	1.49548	1.49812	48.21	.2	1.53134	1.53405	50.48
.3	1.49619	1.49883	48.26	.3	1.53207	1.53478	50.52
.4	1.49690	1.49954	48.30	.4	1.53279	1.53551	50.57
92.5	1.49761	1.50026	48.35	97.5	1.53352	1.53623	50.61
.6	1.49832	1.50097	48.40	.6	1.53425	1.53696	50.66
.7	1.49903	1.50168	48.44	.7	1.53498	1.53769	50.70
.8	1.49974	1.50239	48.49	.8	1.53570	1.53842	50.75
.9	1.50045	1.50310	48.53	.9	1.53643	1.53915	50.79
93.0	1.50116	1.50381	48.58	98.0	1.53716	1.53988	50.84
.1	1.50187	1.50453	48.62	.1	1.53789	1.54061	50.88
.2	1.50258	1.50524	48.67	.2	1.53862	1.54134	50.93
.3	1.50329	1.50595	48.72	.3	1.53935	1.54207	50.97
.4	1.50401	1.50667	48.76	.4	1.54008	1.54280	51.02
93.5	1.50472	1.50738	48.81	98.5	1.54081	1.54353	51.06
.6	1.50543	1.50810	48.85	.6	1.54154	1.54426	51.10
.7	1.50615	1.50881	48.90	.7	1.54227	1.54499	51.15
.8	1.50686	1.50952	48.94	.8	1.54300	1.54573	51.19
.9	1.50757	1.51024	48.99	.9	1.54373	1.54646	51.24
94.0	1.50829	1.51096	49.03	99.0	1.54446	1.54719	51.28
.1	1.50900	1.51167	49.08	.1	1.54519	1.54793	51.33
.2	1.50972	1.51239	49.12	.2	1.54593	1.54866	51.37
.3	1.51044	1.51311	49.17	.3	1.54666	1.54939	51.42
.4	1.51115	1.51382	49.22	.4	1.54739	1.55013	51.46
94.5	1.51187	1.51454	49.26	99.5	1.54812	1.55087	51.50
.6	1.51258	1.51526	49.31	.6	1.54886	1.55160	51.55
.7	1.51330	1.51598	49.35	.7	1.54960	1.55234	51.59
.8	1.51402	1.51670	49.40	.8	1.55033	1.55307	51.64
.9	1.51474	1.51742	49.44	.9	1.55106	1.55381	51.68
				100.0	1.55180	1.55454	51.73

TABLE 32

TEMPERATURE CORRECTIONS TO READINGS OF SACCCHAROMETERS (STANDARD AT 20° C.)\*

(This table is calculated using the data on thermal expansion of sugar solutions by Plato,† assuming the instrument to be of Jena 16<sup>m</sup> glass. The table should be used with caution and only for approximate results when the temperature differs much from the standard temperature or from the temperature of the surrounding air.)

Temperature ° C.	Observed Per Cent of Sugar										
	0	5	10	15	20	25	30	35	40	45	50
0	0.30	0.49	0.65	0.77	0.80	0.99	1.08	1.16	1.24	1.31	1.37
5	.36	.47	.56	.65	.73	.80	.86	.91	.97	1.01	1.05
10	.32	.38	.43	.48	.52	.57	.60	.64	.67	.70	.72
11	.31	.35	.40	.44	.48	.51	.55	.58	.60	.63	.65
12	.29	.32	.36	.40	.43	.46	.50	.52	.54	.56	.58
13	.26	.29	.32	.35	.38	.41	.44	.46	.48	.49	.51
14	.24	.26	.29	.31	.34	.36	.38	.40	.41	.42	.44
15	.20	.22	.24	.26	.28	.30	.32	.33	.34	.36	.36
16	.17	.18	.20	.22	.23	.25	.26	.27	.28	.28	.29
17	.13	.14	.15	.16	.18	.19	.20	.20	.21	.21	.22
18	.09	.10	.10	.11	.12	.13	.13	.14	.14	.14	.15
19	.05	.05	.05	.06	.06	.06	.07	.07	.07	.07	.08

	Subtract from Observed Per Cent										
	0	5	10	15	20	25	30	35	40	45	50
0	0.30	0.49	0.65	0.77	0.80	0.99	1.08	1.16	1.24	1.31	1.37
5	.36	.47	.56	.65	.73	.80	.86	.91	.97	1.01	1.05
10	.32	.38	.43	.48	.52	.57	.60	.64	.67	.70	.72
11	.31	.35	.40	.44	.48	.51	.55	.58	.60	.63	.65
12	.29	.32	.36	.40	.43	.46	.50	.52	.54	.56	.58
13	.26	.29	.32	.35	.38	.41	.44	.46	.48	.49	.51
14	.24	.26	.29	.31	.34	.36	.38	.40	.41	.42	.44
15	.20	.22	.24	.26	.28	.30	.32	.33	.34	.36	.36
16	.17	.18	.20	.22	.23	.25	.26	.27	.28	.28	.29
17	.13	.14	.15	.16	.18	.19	.20	.20	.21	.21	.22
18	.09	.10	.10	.11	.12	.13	.13	.14	.14	.14	.15
19	.05	.05	.05	.06	.06	.06	.07	.07	.07	.07	.08

\* From Circular No. 19, 1914, U. S. Bureau of Standards, p. 25.

† Wiss. Abh. der Kaiserlichen Normal-Eichungs-Kommission, 2, p. 140, 1900.

TABLE 32—Continued  
TEMPERATURE CORRECTIONS TO READINGS OF SACCHAROMETERS

Temperature ° C.	Observed Per Cent of Sugar													
	0	5	10	15	20	25	30	35	40	45	50	55	60	70
	Add to Observed Per Cent													
21	0.04	.05	0.06	0.06	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.09
22	.10	.10	.11	.12	.12	.13	.14	.14	.14	.15	.16	.16	.16	.16
23	.16	.16	.17	.17	.19	.20	.21	.21	.22	.23	.24	.24	.24	.24
24	.21	.22	.23	.24	.26	.27	.28	.29	.30	.31	.32	.32	.32	.32
25	.27	.28	.30	.31	.32	.34	.35	.36	.38	.38	.39	.39	.40	.39
26	.33	.34	.36	.37	.40	.40	.42	.44	.46	.47	.47	.48	.48	.48
27	.40	.41	.42	.44	.46	.48	.50	.52	.54	.54	.55	.56	.56	.56
28	.46	.47	.49	.51	.54	.56	.58	.60	.61	.62	.63	.64	.64	.64
29	.54	.55	.56	.59	.61	.63	.66	.68	.70	.70	.71	.72	.72	.72
30	.61	.62	.63	.66	.68	.70	.73	.76	.78	.78	.79	.80	.80	.81
35	.99	1.01	1.02	1.06	1.10	1.13	1.16	1.18	1.20	1.21	1.22	1.22	1.23	1.22
40	1.42	1.45	1.47	1.51	1.54	1.57	1.62	1.62	1.64	1.65	1.65	1.65	1.66	1.65
45	1.91	1.94	1.96	2.00	2.03	2.05	2.07	2.09	2.10	2.10	2.10	2.10	2.10	2.08
50	2.46	2.48	2.50	2.53	2.56	2.57	2.58	2.59	2.59	2.58	2.58	2.57	2.56	2.52
55	3.05	3.07	3.09	3.12	3.12	3.12	3.12	3.11	3.10	3.08	3.07	3.05	3.03	2.97
60	3.69	3.72	3.73	3.73	3.72	3.70	3.67	3.65	3.62	3.60	3.57	3.54	3.50	3.43

TABLE 33

SHOWING THE WEIGHT PER CUBIC FOOT AND U. S. GALLON (231 Cu. In.) AND SOLIDS (Brix) OF SUGAR SOLUTIONS AT 17  $\frac{1}{2}$ ° C.

(Based upon Stammer's Table, p. 451. Recalculated 1928 to Water Weight of 62.28 pounds per cubic foot at 17  $\frac{1}{2}$ ° C.)

De- gree Brix	Weight of		Solids (Brix) per		De- gree Brix	Weight of		Solids (Brix) per	
	1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
	Lbs.	Lbs.	Lbs.	Lb.		Lbs.	Lbs.	Lbs.	Lbs.
1.0	62.52	8.36	0.63	0.08	11.0	65.04	8.70	7.15	0.96
.2	62.57	8.36	0.75	0.10	.2	65.09	8.71	7.29	0.97
.4	62.62	8.37	0.88	0.12	.4	65.14	8.72	7.43	0.99
.6	62.67	8.38	1.00	0.14	.6	65.20	8.72	7.56	1.01
.8	62.72	8.38	1.13	0.16	.8	65.25	8.73	7.70	1.03
2.0	62.77	8.39	1.26	0.17	12.0	65.30	8.74	7.84	1.05
.2	62.81	8.40	1.38	0.18	.2	65.36	8.74	7.91	1.06
.4	62.86	8.41	1.51	0.20	.4	65.41	8.74	8.11	1.08
.6	62.91	8.42	1.64	0.22	.6	65.46	8.75	8.25	1.10
.8	62.96	8.42	1.76	0.24	.8	65.51	8.76	8.39	1.12
3.0	63.01	8.43	1.89	0.25	13.0	65.57	8.77	8.52	1.14
.2	63.06	8.44	2.02	0.27	.2	65.62	8.77	8.66	1.16
.4	63.11	8.44	2.15	0.29	.4	65.67	8.78	8.80	1.18
.6	63.16	8.45	2.27	0.30	.6	65.73	8.79	8.94	1.20
.8	63.21	8.46	2.40	0.32	.8	65.78	8.80	9.08	1.21
4.0	63.26	8.46	2.53	0.34	14.0	65.83	8.80	9.22	1.23
.2	63.31	8.47	2.66	0.36	.2	65.89	8.81	9.36	1.25
.4	63.36	8.48	2.79	0.37	.4	65.94	8.82	9.50	1.27
.6	63.41	8.48	2.92	0.39	.6	65.99	8.82	9.63	1.29
.8	63.46	8.49	3.05	0.41	.8	66.05	8.83	9.78	1.31
5.0	63.51	8.50	3.18	0.43	15.0	66.10	8.84	9.92	1.33
.2	63.56	8.50	3.31	0.44	.2	66.15	8.84	10.05	1.34
.4	63.61	8.51	3.43	0.46	.4	66.21	8.85	10.20	1.36
.6	63.66	8.52	3.56	0.48	.6	66.26	8.86	10.34	1.38
.8	63.71	8.52	3.70	0.49	.8	66.32	8.87	10.48	1.40
6.0	63.76	8.53	3.83	0.51	16.0	66.37	8.87	10.62	1.42
.2	63.81	8.54	3.96	0.53	.2	66.42	8.88	10.76	1.44
.4	63.86	8.54	4.09	0.55	.4	66.48	8.89	10.90	1.46
.6	63.91	8.55	4.22	0.56	.6	66.53	8.89	11.04	1.48
.8	63.96	8.56	4.35	0.58	.8	66.59	8.90	11.19	1.50
7.0	64.01	8.56	4.48	0.60	17.0	66.64	8.91	11.33	1.51
.2	64.06	8.57	4.61	0.62	.2	66.70	8.92	11.47	1.53
.4	64.11	8.58	4.74	0.63	.4	66.75	8.92	11.61	1.55
.6	64.16	8.58	4.88	0.65	.6	66.80	8.93	11.76	1.57
.8	64.21	8.59	5.01	0.67	.8	66.86	8.94	11.90	1.59
8.0	64.26	8.60	5.14	0.69	18.0	66.91	8.94	12.04	1.61
.2	64.32	8.61	5.27	0.70	.2	66.97	8.95	12.19	1.63
.4	64.37	8.61	5.41	0.72	.4	67.02	8.96	12.33	1.65
.6	64.42	8.62	5.54	0.74	.6	67.08	8.97	12.48	1.67
.8	64.47	8.63	5.67	0.76	.8	67.13	8.97	12.62	1.69
9.0	64.52	8.63	5.81	0.78	19.0	67.19	8.98	12.77	1.71
.2	64.57	8.64	5.94	0.79	.2	67.25	8.99	12.91	1.73
.4	64.62	8.65	6.07	0.81	.4	67.30	9.00	13.06	1.75
.6	64.68	8.65	6.21	0.83	.6	67.36	9.01	13.20	1.76
.8	64.73	8.66	6.34	0.85	.8	67.41	9.01	13.34	1.78
10.0	64.78	8.67	6.48	0.87	20.0	67.47	9.02	13.49	1.80
.2	64.83	8.67	6.61	0.88	.2	67.52	9.03	13.64	1.82
.4	64.88	8.68	6.75	0.90	.4	67.58	9.03	13.79	1.84
.6	64.94	8.69	6.88	0.92	.6	67.64	9.04	13.93	1.86
.8	64.99	8.70	7.02	0.94	.8	67.69	9.05	14.08	1.88

TABLE 33—Continued

SHOWING THE WEIGHT PER CUBIC FOOT AND U. S. GALLON (231 Cu. In.) AND SOLIDS (Brix) OF SUGAR SOLUTIONS AT 17½° C.

(Based upon Stammer's Table, p. 451. Recalculated 1928 to Water Weight of 62.28 pounds per cubic foot at 17½° C.)

De- gree Brix	Weight of		Solids (Brix) per		De- gree Brix	Weight of		Solids (Brix) per	
	1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
<b>21.0</b>	Lbs.	Lbs.	Lbs.	Lbs.	<b>31.0</b>	Lbs.	Lbs.	Lbs.	Lbs.
.2	67.75	9.06	14.23	1.90	.2	70.66	9.45	21.90	2.93
.4	67.80	9.06	14.37	1.92	.4	70.72	9.45	22.06	2.95
.6	67.86	9.07	14.52	1.94	.6	70.78	9.46	22.22	2.97
.8	67.92	9.08	14.67	1.96	.8	70.84	9.47	22.39	2.99
	67.97	9.09	14.82	1.98		70.90	9.48	22.55	3.01
<b>22.0</b>	68.03	9.09	14.97	2.00	<b>32.0</b>	70.96	9.49	22.71	3.04
.2	68.09	9.10	15.12	2.02	.2	71.02	9.49	22.87	3.06
.4	68.14	9.11	15.26	2.04	.4	71.08	9.50	23.03	3.08
.6	68.20	9.12	15.41	2.06	.6	71.14	9.51	23.19	3.10
.8	68.26	9.12	15.56	2.08	.8	71.20	9.52	23.35	3.12
<b>23.0</b>	68.31	9.13	15.71	2.10	<b>33.0</b>	71.26	9.53	23.52	3.14
.2	68.37	9.14	15.86	2.12	.2	71.32	9.53	23.68	3.17
.4	68.43	9.15	16.01	2.14	.4	71.39	9.54	23.84	3.19
.6	68.48	9.15	16.16	2.16	.6	71.45	9.55	24.01	3.21
.8	68.54	9.16	16.31	2.18	.8	71.51	9.56	24.17	3.23
<b>24.0</b>	68.60	9.17	16.46	2.20	<b>34.0</b>	71.57	9.57	24.33	3.25
.2	68.66	9.18	16.62	2.22	.2	71.63	9.58	24.50	3.28
.4	68.71	9.18	16.77	2.24	.4	71.69	9.58	24.66	3.30
.6	68.77	9.19	16.92	2.26	.6	71.75	9.59	24.83	3.32
.8	68.83	9.20	17.07	2.28	.8	71.82	9.60	24.99	3.34
<b>25.0</b>	68.89	9.21	17.22	2.30	<b>35.0</b>	71.88	9.61	25.16	3.36
.2	68.94	9.22	17.37	2.32	.2	71.94	9.62	25.32	3.39
.4	69.00	9.22	17.53	2.34	.4	72.00	9.63	25.49	3.41
.6	69.06	9.23	17.68	2.36	.6	72.06	9.63	25.65	3.42
.8	69.12	9.24	17.83	2.38	.8	72.13	9.64	25.82	3.45
<b>26.0</b>	69.18	9.25	17.99	2.41	<b>36.0</b>	72.19	9.65	25.99	3.47
.2	69.23	9.25	18.14	2.43	.2	72.25	9.66	26.15	3.50
.4	69.29	9.26	18.29	2.45	.4	72.31	9.67	26.32	3.52
.6	69.35	9.27	18.45	2.47	.6	72.38	9.68	26.49	3.54
.8	69.41	9.28	18.60	2.49	.8	72.44	9.69	26.66	3.56
<b>27.0</b>	69.47	9.28	18.76	2.51	<b>37.0</b>	72.50	9.69	26.83	3.59
.2	69.53	9.29	18.91	2.53	.2	72.56	9.70	26.99	3.61
.4	69.58	9.30	19.06	2.55	.4	72.63	9.71	27.16	3.63
.6	69.64	9.31	19.22	2.57	.6	72.69	9.72	27.33	3.65
.8	69.70	9.32	19.38	2.59	.8	72.75	9.73	27.50	3.68
<b>28.0</b>	69.76	9.32	19.53	2.61	<b>38.0</b>	72.82	9.74	27.67	3.70
.2	69.82	9.33	19.69	2.63	.2	72.88	9.74	27.84	3.72
.4	69.88	9.34	19.85	2.65	.4	72.94	9.75	28.01	3.74
.6	69.94	9.35	20.00	2.67	.6	73.01	9.76	28.18	3.77
.8	70.00	9.36	20.16	2.70	.8	73.07	9.77	28.35	3.79
<b>29.0</b>	70.06	9.36	20.32	2.72	<b>39.0</b>	73.14	9.78	28.52	3.81
.2	70.12	9.37	20.48	2.74	.2	73.20	9.79	28.69	3.84
.4	70.18	9.38	20.63	2.76	.4	73.26	9.80	28.86	3.86
.6	70.24	9.39	20.79	2.78	.6	73.33	9.80	29.04	3.88
.8	70.30	9.40	20.95	2.80	.8	73.39	9.81	29.21	3.91
<b>30.0</b>	70.36	9.40	21.11	2.82	<b>40.0</b>	73.45	9.82	29.38	3.93
.2	70.42	9.41	21.27	2.84	.2	73.52	9.83	29.56	3.95
.4	70.48	9.42	21.43	2.86	.4	73.58	9.84	29.73	3.97
.6	70.54	9.43	21.59	2.89	.6	73.65	9.85	29.90	4.00
.8	70.60	9.44	21.74	2.91	.8	73.71	9.86	30.07	4.02



TABLE 33—Continued

SHOWING THE WEIGHT PER CUBIC FOOT AND U. S. GALLON (231 Cu. In.) AND SOLIDS (Brix) OF SUGAR SOLUTIONS AT 17½° C.

(Based upon Stammer's Table, p. 451. Recalculated 1928 to Water Weight of 62.28 pounds per cubic foot at 17½° C.)

De- gree Brix	Weight of		Solids (Brix) per		De- gree Brix	Weight of		Solids (Brix) per	
	1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
	Lbs.	Lbs.	Lbs.	Lbs.		Lbs.	Lbs.	Lbs.	Lbs.
41.0	73.78	9.86	30.25	4.04	51.0	77.12	10.31	39.33	5.26
.2	73.84	9.87	30.42	4.07	.2	77.19	10.32	39.52	5.28
.4	73.91	9.88	30.60	4.09	.4	77.26	10.33	39.71	5.31
.6	73.97	9.89	30.77	4.11	.6	77.33	10.34	39.90	5.33
.8	74.04	9.90	30.95	4.14	.8	77.40	10.35	40.09	5.36
42.0	74.10	9.91	31.12	4.16	52.0	77.47	10.36	40.28	5.38
.2	74.17	9.92	31.30	4.18	.2	77.54	10.37	40.48	5.41
.4	74.23	9.93	31.47	4.21	.4	77.61	10.38	40.67	5.44
.6	74.30	9.93	31.65	4.23	.6	77.68	10.38	40.86	5.46
.8	74.36	9.94	31.83	4.26	.8	77.75	10.39	41.05	5.49
43.0	74.43	9.95	32.00	4.28	53.0	77.82	10.40	41.24	5.51
.2	74.49	9.96	32.18	4.30	.2	77.89	10.41	41.44	5.54
.4	74.56	9.97	32.36	4.33	.4	77.96	10.42	41.63	5.57
.6	74.63	9.98	32.54	4.35	.6	78.03	10.43	41.82	5.59
.8	74.69	9.98	32.71	4.37	.8	78.10	10.44	42.02	5.62
44.0	74.76	9.99	32.89	4.40	54.0	78.17	10.45	42.21	5.64
.2	74.82	10.00	33.07	4.42	.2	78.24	10.46	42.41	5.67
.4	74.89	10.01	33.25	4.45	.4	78.32	10.47	42.61	5.70
.6	74.96	10.02	33.43	4.47	.6	78.38	10.48	42.80	5.72
.8	75.02	10.03	33.61	4.49	.8	78.45	10.49	42.99	5.75
45.0	75.09	10.04	33.79	4.52	55.0	78.53	10.50	43.19	5.77
.2	75.15	10.05	33.97	4.54	.2	78.60	10.51	43.39	5.80
.4	75.22	10.06	34.15	4.57	.4	78.67	10.52	43.58	5.83
.6	75.29	10.07	34.33	4.59	.6	78.74	10.53	43.78	5.85
.8	75.35	10.07	34.51	4.61	.8	78.81	10.54	43.98	5.88
46.0	75.42	10.08	34.69	4.64	56.0	78.88	10.55	44.17	5.91
.2	75.49	10.09	34.88	4.66	.2	78.95	10.55	44.37	5.93
.4	75.55	10.10	35.06	4.69	.4	79.03	10.57	44.57	5.96
.6	75.62	10.11	35.24	4.71	.6	79.10	10.57	44.77	5.99
.8	75.69	10.12	35.42	4.74	.8	79.17	10.58	44.97	6.01
47.0	75.76	10.13	35.61	4.76	57.0	79.24	10.59	45.17	6.04
.2	75.82	10.14	35.79	4.78	.2	79.31	10.60	45.37	6.07
.4	75.89	10.15	35.97	4.81	.4	79.38	10.61	45.56	6.09
.6	75.96	10.16	36.16	4.83	.6	79.46	10.62	45.77	6.12
.8	76.03	10.16	36.34	4.86	.8	79.53	10.63	45.97	6.15
48.0	76.09	10.17	36.52	4.88	58.0	79.60	10.64	46.17	6.17
.2	76.16	10.18	36.71	4.91	.2	79.68	10.65	46.37	6.20
.4	76.23	10.19	36.90	4.93	.4	79.75	10.66	46.57	6.23
.6	76.30	10.20	37.08	4.96	.6	79.82	10.67	46.77	6.25
.8	76.37	10.21	37.27	4.98	.8	79.89	10.68	46.98	6.28
49.0	76.43	10.22	37.45	5.01	59.0	79.97	10.69	47.18	6.31
.2	76.50	10.23	37.64	5.03	.2	80.04	10.70	47.38	6.33
.4	76.57	10.24	37.83	5.06	.4	80.11	10.71	47.59	6.36
.6	76.64	10.25	38.01	5.08	.6	80.19	10.72	47.79	6.39
.8	76.71	10.26	38.20	5.11	.8	80.27	10.73	48.00	6.42
50.0	76.78	10.26	38.39	5.13	60.0	80.33	10.74	48.20	6.44
.2	76.85	10.27	38.58	5.16	.2	80.41	10.75	48.41	6.47
.4	76.92	10.28	38.77	5.18	.4	80.48	10.76	48.61	6.50
.6	76.98	10.29	38.95	5.21	.6	80.55	10.77	48.81	6.53
.8	77.05	10.30	39.14	5.23	.8	80.63	10.78	49.02	6.55

TABLE 33—Continued

SHOWING THE WEIGHT PER CUBIC FOOT AND U. S. GALLON (231 Cu. In.) AND SOLIDS (Brix) OF SUGAR SOLUTIONS AT 17½° C.

(Based upon Stammer's Table, p. 451. Recalculated 1928 to Water Weight of 62.28 pounds per cubic foot at 17½° C.)

De- gree Brix	Weight of		Solids (Brix) per		De- gree Brix	Weight of		Solids (Brix) per	
	1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
	Lbs.	Lbs.	Lbs.	Lbs.		Lbs.	Lbs.	Lbs.	Lbs.
61.0	80.70	10.79	49.23	6.58	71.0	84.53	11.80	60.02	8.02
.2	80.78	10.80	49.44	6.61	.2	84.61	11.81	60.24	8.05
.4	80.85	10.81	49.64	6.64	.4	84.68	11.82	60.46	8.08
.6	80.93	10.82	49.85	6.66	.6	84.76	11.83	60.69	8.11
.8	81.00	10.83	50.06	6.69	.8	84.84	11.84	60.92	8.14
62.0	81.07	10.84	50.26	6.72	72.0	84.92	11.85	61.14	8.17
.2	81.15	10.85	50.48	6.75	.2	85.00	11.86	61.37	8.20
.4	81.22	10.86	50.68	6.78	.4	85.08	11.87	61.60	8.24
.6	81.30	10.87	50.89	6.80	.6	85.16	11.88	61.83	8.27
.8	81.37	10.88	51.10	6.83	.8	85.24	11.40	62.05	8.30
63.0	81.45	10.89	51.31	6.86	73.0	85.32	11.41	62.28	8.33
.2	81.52	10.90	51.52	6.89	.2	85.40	11.42	62.51	8.36
.4	81.60	10.91	51.73	6.92	.4	85.48	11.43	62.74	8.39
.6	81.67	10.92	51.94	6.94	.6	85.56	11.44	62.97	8.42
.8	81.75	10.93	52.16	6.97	.8	85.64	11.45	63.20	8.45
64.0	81.82	10.94	52.36	7.00	74.0	85.72	11.46	63.43	8.48
.2	81.90	10.95	52.58	7.03	.2	85.80	11.47	63.66	8.51
.4	81.98	10.96	52.80	7.06	.4	85.88	11.48	63.89	8.54
.6	82.05	10.97	53.00	7.09	.6	85.96	11.49	64.13	8.57
.8	82.13	10.98	53.22	7.11	.8	86.04	11.50	64.36	8.60
65.0	82.20	10.99	52.43	7.14	75.0	86.13	11.51	64.60	8.64
.2	82.28	11.00	53.65	7.17	.2	86.21	11.53	64.83	8.67
.4	82.35	11.01	53.86	7.20	.4	86.29	11.54	65.06	8.70
.6	82.43	11.02	54.07	7.23	.6	86.37	11.55	65.30	8.73
.8	82.51	11.03	54.29	7.26	.8	86.45	11.56	65.53	8.76
66.0	82.58	11.04	54.50	7.29	76.0	86.53	11.57	65.76	8.79
.2	82.66	11.05	54.72	7.32	.2	86.61	11.58	66.00	8.82
.4	82.74	11.06	54.94	7.34	.4	86.69	11.59	66.23	8.85
.6	82.81	11.07	55.15	7.37	.6	86.78	11.60	66.47	8.89
.8	82.89	11.08	55.37	7.40	.8	86.86	11.61	66.70	8.92
67.0	82.97	11.09	55.59	7.43	77.0	86.94	11.62	66.94	8.95
.2	83.04	11.10	55.80	7.46	.2	87.02	11.63	67.18	8.98
.4	83.12	11.11	56.02	7.49	.4	87.10	11.64	67.42	9.01
.6	83.20	11.12	56.24	7.52	.6	87.19	11.66	67.66	9.05
.8	83.28	11.13	56.46	7.55	.8	87.27	11.67	67.90	9.08
68.0	83.35	11.14	56.68	7.58	78.0	87.35	11.68	68.13	9.11
.2	83.43	11.15	56.90	7.61	.2	87.43	11.69	69.37	9.14
.4	83.51	11.16	57.12	7.64	.4	87.52	11.70	68.62	9.17
.6	83.59	11.18	57.34	7.67	.6	87.60	11.71	68.85	9.20
.8	83.66	11.18	57.56	7.70	.8	87.68	11.72	69.09	9.24
69.0	83.73	11.19	57.77	7.72	79.0	87.76	11.73	69.37	9.27
.2	83.82	11.21	58.00	7.75	.2	87.85	11.74	69.58	9.30
.4	83.90	11.22	58.23	7.78	.4	87.93	11.76	69.82	9.33
.6	83.98	11.23	58.45	7.81	.6	88.01	11.77	70.06	9.37
.8	84.05	11.24	58.67	7.84	.8	88.10	11.78	70.30	9.40
70.0	84.12	11.25	58.88	7.87	80.0	88.18	11.79	70.54	9.43
.2	84.21	11.26	59.12	7.90	.2	88.26	11.80	70.78	9.46
.4	84.29	11.27	59.34	7.93	.4	88.35	11.81	71.03	9.50
.6	84.37	11.28	59.57	7.96	.6	88.43	11.82	71.27	9.53
.8	84.45	11.29	59.79	7.99	.8	88.51	11.83	71.52	9.56

TABLE 33—Continued

SHOWING THE WEIGHT PER CUBIC FOOT AND U. S. GALLON (231 Cu. In.) AND SOLIDS (Brix) OF SUGAR SOLUTIONS AT 17  $\frac{1}{2}$ ° C.

(Based upon Stammer's Table, p. 451. Recalculated 1928 to Water Weight of 62.28 pounds per cubic foot at 17  $\frac{1}{2}$ ° C.)

De- gree Brix	Weight of		Solids (Brix) per		De- gree Brix	Weight of		Solids (Brix) per	
	1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.		1 Cu. Ft.	1 Gal.	1 Cu. Ft.	1 Gal.
<b>81.0</b>	Lbs.	Lbs.	Lbs.	Lbs.	<b>89.0</b>	Lbs.	Lbs.	Lbs.	Lbs.
.2	88.60	11.84	71.77	9.59	.2	92.04	12.30	81.92	10.95
.4	88.68	11.86	72.01	9.63	.4	92.12	12.32	82.17	10.99
.6	88.77	11.87	72.26	9.66	.6	92.21	12.33	82.44	11.02
.8	88.85	11.88	72.50	9.69	.8	92.30	12.34	82.70	11.06
	88.93	11.89	72.74	9.72		92.39	12.35	82.97	11.09
<b>82.0</b>	89.02	11.90	73.00	9.76	<b>90.0</b>	92.48	12.36	83.23	11.13
.2	89.10	11.91	73.24	9.79	.2	92.57	12.38	83.50	11.16
.4	89.19	11.92	73.49	9.82	.4	92.65	12.39	83.76	11.20
.6	89.27	11.93	73.74	9.86	.6	92.74	12.40	84.02	11.23
.8	89.36	11.95	73.99	9.89	.8	92.83	12.41	84.29	11.27
<b>83.0</b>	89.44	11.96	74.24	9.93	<b>91.0</b>	92.92	12.42	84.56	11.30
.2	89.53	11.97	74.49	9.96	.2	93.01	12.43	84.83	11.34
.4	89.61	11.98	74.73	9.99	.4	93.10	12.45	85.09	11.38
.6	89.70	11.99	74.99	10.03	.6	93.19	12.46	85.36	11.41
.8	89.78	12.00	75.24	10.06	.8	93.28	12.47	85.63	11.45
<b>84.0</b>	89.87	12.01	75.49	10.09	<b>92.0</b>	93.37	12.48	85.90	11.48
.2	89.95	12.03	75.74	10.13	.2	93.46	12.49	86.17	11.52
.4	90.04	12.04	75.99	10.16	.4	93.55	12.50	86.44	11.56
.6	90.13	12.05	76.25	10.19	.6	93.64	12.52	86.71	11.59
.8	90.21	12.06	76.50	10.23	.8	93.73	12.53	86.98	11.63
<b>85.0</b>	90.30	12.07	76.76	10.26	<b>93.0</b>	93.82	12.54	87.25	11.66
.2	90.38	12.08	77.00	10.29	.2	93.91	12.55	87.52	11.70
.4	90.47	12.09	77.26	10.33	.4	94.00	12.57	87.80	11.74
.6	90.56	12.11	77.52	10.36	.6	94.09	12.58	88.07	11.77
.8	90.64	12.12	77.77	10.40	.8	94.18	12.59	88.34	11.81
<b>86.0</b>	90.73	12.13	78.03	10.43	<b>94.0</b>	94.27	12.60	88.60	11.84
.2	90.81	12.14	78.28	10.47	.2	94.36	12.61	88.89	11.88
.4	90.90	12.15	78.54	10.50	.4	94.45	12.63	89.15	11.92
.6	90.99	12.16	78.80	10.53	.6	94.54	12.64	89.43	11.96
.8	91.08	12.18	79.06	10.57	.8	94.63	12.65	89.71	11.99
<b>87.0</b>	91.16	12.19	79.31	10.60	<b>95.0</b>	94.72	12.66	89.98	12.03
.2	91.25	12.20	79.57	10.64	.2	94.81	12.68	90.26	12.07
.4	91.34	12.21	79.83	10.67	.4	94.90	12.69	90.53	12.10
.6	91.42	12.22	80.08	10.71	.6	94.99	12.70	90.81	12.14
.8	91.51	12.23	80.34	10.74	.8	95.08	12.71	91.09	12.18
<b>88.0</b>	91.60	12.25	80.61	10.78	<b>96.0</b>	95.17	12.72	91.36	12.21
.2	91.69	12.26	80.87	10.81	.2	95.26	12.74	91.64	12.25
.4	91.77	12.27	81.12	10.84	.4	95.35	12.75	91.92	12.29
.6	91.86	12.28	81.39	10.88	.6	95.44	12.76	92.20	12.33
.8	91.95	12.29	81.65	10.92	.8	95.53	12.77	92.47	12.36

TABLE 33A

WEIGHTS PER U. S. GALLON AND WEIGHTS PER CUBIC FOOT OF SUGAR (SUCROSE) SOLUTIONS AT 20° C.

This table is based on the density value of Plato for solutions of cane sugar. The Baumé values are from the table of Bates and Bearce. The weights are for brass weights, density 8.4. One U. S. gallon, 231 cubic inches. One pound, av. 453.5924 grams. One U. S. gallon of water weighs 3778.649 grams (8.33049 pounds av.) in vacuo.

Per Cent Sucrose by Weight (Brix)	Weight per Gallon in Air		Weight per Gallon in Vacuo		Weight per Cubic Foot in Air Pounds	Specific Gravity 20°/4° C.	Specific Gravity 20°/20° C.	Baumé = $\frac{145}{145 - \text{Sp. Gr. } 20^\circ/20^\circ \text{C.}}$
	Lbs.	Gms.	Lbs.	Gms.				
0	8.322	3774.6	8.330	3778.6	62.25	0.99823	1.00000	0.00
1	8.354	3789.3	8.363	3793.3	62.49	1.00212	1.00389	0.56
2	8.387	3804.1	8.395	3808.1	62.74	1.00602	1.00779	1.12
3	8.419	3818.9	8.428	3822.9	62.98	1.00993	1.01172	1.68
4	8.452	3833.9	8.461	3837.9	63.23	1.01388	1.01567	2.24
5	8.485	3848.9	8.494	3852.9	63.48	1.01785	1.01965	2.79
6	8.519	3864.1	8.528	3868.1	63.73	1.02186	1.02366	3.35
7	8.552	3879.3	8.561	3883.3	63.98	1.02588	1.02770	3.91
8	8.586	3894.7	8.595	3898.7	64.23	1.02994	1.03176	4.46
9	8.620	3910.2	8.629	3914.2	64.49	1.03403	1.03586	5.02
10	8.655	3925.7	8.664	3929.7	64.74	1.03814	1.03998	5.57
11	8.689	3941.4	8.698	3945.4	65.00	1.04229	1.04413	6.13
12	8.724	3957.2	8.733	3961.2	65.26	1.04646	1.04831	6.68
13	8.759	3973.1	8.768	3977.1	65.52	1.05066	1.05252	7.24
14	8.795	3989.2	8.803	3993.2	65.79	1.05490	1.05677	7.79
15	8.830	4005.3	8.839	4009.3	66.05	1.05916	1.06104	8.34
16	8.866	4021.6	8.875	4025.6	66.32	1.06346	1.06534	8.89
17	8.902	4038.0	8.911	4041.9	66.59	1.06779	1.06968	9.45
18	8.939	4054.5	8.947	4058.4	66.87	1.07215	1.07404	10.00
19	8.975	4071.1	8.984	4075.0	67.14	1.07654	1.07844	10.55
20	9.012	4087.8	9.021	4091.8	67.42	1.08096	1.08287	11.10
21	9.049	4104.7	9.058	4108.6	67.69	1.08541	1.08733	11.65
22	9.087	4121.7	9.095	4125.6	67.97	1.08990	1.09183	12.20
23	9.125	4138.8	9.133	4142.8	68.26	1.09442	1.09636	12.74
24	9.163	4156.0	9.171	4160.0	68.54	1.09897	1.10092	13.29
25	9.201	4173.4	9.209	4177.3	68.83	1.10356	1.10551	13.84
26	9.239	4190.9	9.248	4194.8	69.11	1.10818	1.11014	14.39
27	9.278	4208.5	9.287	4212.4	69.41	1.11283	1.11480	14.93
28	9.317	4226.2	9.326	4230.2	69.70	1.11751	1.11949	15.48
29	9.357	4244.1	9.365	4248.0	69.99	1.12223	1.12422	16.02
30	9.396	4262.1	9.405	4266.0	70.29	1.12698	1.12898	16.57
31	9.436	4280.2	9.445	4284.2	70.59	1.13177	1.13378	17.11
32	9.477	4298.5	9.485	4302.4	70.89	1.13660	1.13861	17.65
33	9.517	4316.8	9.526	4320.8	71.19	1.14145	1.14347	18.19
34	9.558	4335.4	9.566	4339.3	71.50	1.14634	1.14837	18.73
35	9.599	4354.0	9.608	4358.0	71.81	1.15128	1.15331	19.28
36	9.640	4372.8	9.649	4376.7	72.12	1.15624	1.15828	19.81
37	9.682	4391.8	9.691	4395.7	72.43	1.16124	1.16329	20.35
38	9.724	4410.8	9.733	4414.7	72.74	1.16627	1.16833	20.89
39	9.766	4430.0	9.775	4433.9	73.06	1.17134	1.17341	21.43
40	9.809	4449.3	9.818	4453.3	73.38	1.17645	1.17853	21.97
41	9.852	4468.8	9.861	4472.7	73.70	1.18159	1.18368	22.50
42	9.895	4488.4	9.904	4492.3	74.02	1.18677	1.18887	23.04
43	9.939	4508.2	9.947	4512.1	74.35	1.19199	1.19410	23.57
44	9.983	4528.1	9.991	4532.0	74.68	1.19725	1.19936	24.10
45	10.027	4548.1	10.035	4552.0	75.01	1.20254	1.20467	24.63
46	10.071	4568.3	10.080	4572.2	75.34	1.20787	1.21001	25.17
47	10.116	4588.6	10.125	4592.5	75.67	1.21324	1.21538	25.70
48	10.161	4609.1	10.170	4613.0	76.01	1.21864	1.22080	26.23
49	10.207	4629.7	10.215	4633.6	76.35	1.22409	1.22625	26.75

TABLE 33A—Continued

WEIGHTS PER U. S. GALLON AND WEIGHTS PER CUBIC FOOT OF SUGAR (SUCROSE) SOLUTIONS AT 20° C.

Per Cent Sucrose by Weight (Brix)	Weight per Gallon in Air		Weight per Gallon in Vacuo		Weight per Cubic Foot in Air Pounds	Specific Gravity 20°/4° C.	Specific Gravity 20°/20° C.	Baumé = 145 - Sp. Gr. 20°/20°
	Lbs.	Gms.	Lbs.	Gms.				
50	10.252	4650.4	10.261	4654.3	76.69	1.22957	1.23174	27.28
51	10.299	4671.3	10.307	4675.2	77.04	1.23508	1.23727	27.81
52	10.345	4692.4	10.353	4696.3	77.39	1.24064	1.24284	28.33
53	10.392	4713.5	10.400	4717.4	77.73	1.24623	1.24844	28.86
54	10.439	4734.9	10.447	4738.7	78.09	1.25187	1.25408	29.38
55	10.486	4756.3	10.494	4760.2	78.44	1.25754	1.25976	29.90
56	10.534	4777.9	10.542	4781.8	78.80	1.26324	1.26548	30.42
57	10.581	4799.7	10.590	4803.5	79.15	1.26899	1.27123	30.94
58	10.630	4821.6	10.638	4825.4	79.52	1.27477	1.27703	31.46
59	10.678	4843.6	10.687	4847.5	79.88	1.28060	1.28286	31.97
60	10.727	4865.8	10.736	4869.7	80.25	1.28646	1.28873	32.49
61	10.777	4888.1	10.785	4892.0	80.61	1.29235	1.29464	33.00
62	10.826	4910.6	10.835	4914.5	80.98	1.29829	1.30059	33.51
63	10.876	4933.2	10.884	4937.1	81.36	1.30427	1.30657	34.02
64	10.926	4956.0	10.935	4959.9	81.73	1.31028	1.31260	34.53
65	10.977	4978.9	10.985	4982.8	82.11	1.31633	1.31866	35.04
66	11.027	5002.2	11.036	5006.8	82.49	1.32242	1.32476	35.55
67	11.079	5025.2	11.087	5029.0	82.87	1.32855	1.33090	36.05
68	11.130	5048.5	11.139	5052.4	83.26	1.33472	1.33708	36.55
69	11.182	5072.0	11.190	5075.9	83.65	1.34093	1.34330	37.06
70	11.234	5095.7	11.242	5099.5	84.04	1.34717	1.34956	37.56
71	11.286	5119.5	11.295	5123.3	84.43	1.35346	1.35585	38.06
72	11.339	5143.4	11.348	5147.2	84.82	1.35978	1.36218	38.55
73	11.392	5167.5	11.401	5171.3	85.22	1.36614	1.36856	39.05
74	11.446	5191.7	11.454	5195.5	85.62	1.37254	1.37496	39.54
75	11.499	5216.1	11.508	5219.9	86.02	1.37897	1.38141	40.05
76	11.554	5240.6	11.562	5244.4	86.43	1.38545	1.38790	40.55
77	11.608	5265.2	11.616	5269.0	86.83	1.39196	1.39442	41.01
78	11.663	5290.0	11.671	5293.8	87.24	1.39850	1.40098	41.50
79	11.717	5315.0	11.726	5318.8	87.65	1.40509	1.40758	41.99
80	11.773	5340.0	11.781	5343.8	88.07	1.41172	1.41421	42.47
81	11.828	5365.2	11.837	5369.0	88.48	1.41837	1.42088	42.95
82	11.884	5390.6	11.893	5394.4	88.90	1.42507	1.42759	43.43
83	11.940	5416.1	11.949	5419.9	89.32	1.43181	1.43434	43.91
84	11.997	5441.7	12.005	5445.5	89.74	1.43858	1.44112	44.38
85	12.054	5467.5	12.062	5471.3	90.17	1.44539	1.44794	44.86
86	12.111	5493.4	12.119	5497.2	90.60	1.45223	1.45480	45.33
87	12.168	5519.5	12.177	5523.3	91.03	1.45911	1.46170	45.80
88	12.226	5545.6	12.234	5549.4	91.46	1.46603	1.46862	46.27
89	12.284	5572.0	12.292	5575.7	91.89	1.47299	1.47559	46.73
90	12.342	5598.4	12.351	5602.2	92.33	1.47998	1.48259	47.20
91	12.401	5625.1	12.409	5628.8	92.77	1.48700	1.48963	47.66
92	12.460	5651.8	12.468	5655.5	93.21	1.49406	1.49671	48.12
93	12.519	5678.6	12.527	5682.4	93.65	1.50116	1.50381	48.58
94	12.579	5705.7	12.587	5709.4	94.10	1.50829	1.51096	49.03
95	12.639	5732.8	12.647	5736.5	94.54	1.51546	1.51814	49.49

Note:—The above table by Snyder and Hammond is included through the Courtesy of the Bureau of Standards.

TABLE 34

GEERLIGS' TABLE FOR DRY SUBSTANCE IN SUGAR-HOUSE PRODUCTS BY ABBÉ  
REFRACTOMETER, AT 28° C.

(Int. Sug. J., 10, p. 69)

Index Refraction	Per Cent Dry Substance	Decimals	Decimals
1.3335	1	0.0001=0.05	0.0010=0.75
1.3349	2	0.0002=0.1	0.0011=0.8
1.3364	3	0.0003=0.2	0.0012=0.8
1.3379	4	0.0004=0.25	0.0013=0.85
1.3394	5	0.0005=0.3	0.0014=0.9
1.3409	6	0.0006=0.4	0.0015=1.0
1.3424	7	0.0007=0.5	
1.3439	8	0.0008=0.6	
1.3454	9	0.0009=0.7	
1.3469	10		
1.3484	11	0.0001=0.05	
1.3500	12	0.0002=0.1	
1.3516	13,	0.0003=0.2	
1.3530	14	0.0004=0.25	
1.3546	15	0.0005=0.3	
1.3562	16 .	0.0006=0.4	
1.3578	17 .	0.0007=0.45	
1.3594	18	0.0008=0.5	
1.3611	19	0.0009=0.6	
1.3627	20	0.0010=0.65	
1.3644	21	0.0011=0.7	
1.3661	22	0.0012=0.75	
1.3678	23	0.0013=0.8	
1.3695	24	0.0014=0.85	
1.3712	25	0.0015=0.9	
1.3729	26	0.0016=0.95	
1.3746	27	0.0001=0.05	0.0012=0.6
1.3764	28	0.0002=0.1	0.0013=0.65
1.3782	29	0.0003=0.15	0.0014=0.7
1.3800	30	0.0004=0.2	0.0015=0.75
1.3818	31	0.0005=0.25	0.0016=0.8
1.3836	32	0.0006=0.3	0.0017=0.85
1.3854	33	0.0007=0.35	0.0018=0.9
1.3872	34	0.0008=0.4	0.0019=0.95
1.3890	35	0.0009=0.45	0.0020=1.0
1.3909	36	0.0010=0.5	0.0021=1.0
1.3928	37	0.0011=0.55	
1.3947	38		
1.3966	39		
1.3984	40		
1.4003	41		

TABLE 34--Continued

GEERLIGS' TABLE FOR DRY SUBSTANCE IN SUGAR-HOUSE PRODUCTS, ETC.

Index	Per Cent Dry Substance	Decimals	Decimals
1.4023	42	0.0001=0.05	0.0012=0.6
1.4043	43	0.0002=0.1	0.0013=0.65
1.4063	44	0.0003=0.15	0.0014=0.7
1.4083	45	0.0004=0.2	0.0015=0.75
1.4104	46	0.0005=0.25	0.0016=0.8
1.4124	47	0.0006=0.3	0.0017=0.85
1.4145	48	0.0007=0.35	0.0018=0.9
1.4166	49	0.0008=0.4	0.0019=0.95
1.4186	50	0.0009=0.45	0.0020=1.0
1.4207	51	0.0010=0.5	0.0021=1.0
1.4228	52	0.0011=0.55	
1.4249	53		
1.4270	54		
1.4292	55	0.0001=0.05	0.0013=0.55
1.4314	56	0.0002=0.1	0.0014=0.6
1.4337	57	0.0003=0.1	0.0015=0.65
1.4359	58	0.0004=0.15	0.0016=0.7
1.4382	59	0.0005=0.2	0.0017=0.75
1.4405	60	0.0006=0.25	0.0018=0.8
1.4428	61	0.0007=0.3	0.0019=0.85
1.4451	62	0.0008=0.35	0.0020=0.9
1.4474	63	0.0009=0.4	0.0021=0.9
1.4497	64	0.0010=0.45	0.0022=0.95
1.4520	65	0.0011=0.5	0.0023=1.0
1.4543	66	0.0012=0.5	0.0024=1.0
1.4567	67		
1.4591	68		
1.4615	69		
1.4639	70		
1.4663	71		
1.4687	72		
1.4711	73	0.0001=0.0	0.0015=0.55
1.4736	74	0.0002=0.05	0.0016=0.6
1.4761	75	0.0003=0.1	0.0017=0.65
1.4786	76	0.0004=0.15	0.0018=0.65
1.4811	77	0.0005=0.2	0.0019=0.7
1.4836	78	0.0006=0.2	0.0020=0.75
1.4862	79	0.0007=0.25	0.0021=0.8
1.4888	80	0.0008=0.3	0.0022=0.8
1.4914	81	0.0009=0.35	0.0023=0.85
1.4940	82	0.0010=0.35	0.0024=0.9
1.4966	83	0.0011=0.4	0.0025=0.9
1.4992	84	0.0012=0.45	0.0026=0.95
1.5019	85	0.0013=0.5	0.0027=1.0
1.5046	86	0.0014=0.5	0.0028=1.0
1.5073	87		
1.5100	88		
1.5127	89		
1.5155	90		





TABLE 36

SCHMITZ'S TABLE (RECALCULATED) FOR SUCROSE FOR USE IN HORNE'S DRY LEAD METHOD AND IN POLARIZING UNDILUTED SOLUTIONS  
(This table is arranged after Schmitz, but no allowance is made for variations in the rotatory power of sucrose. The investigations of Browne \* show such allowance to be unnecessary.)

Pol. Reading	Degrees Brix and Per Cents Sucrose																		Pol. Reading	
	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0		
1	0.26	0.26	0.26	0.26	0.26	0.26	0.26	0.26	0.26	0.26	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	1
2	0.52	0.52	0.52	0.52	0.52	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.50	0.50	0.50	0.50	0.50	2
3	0.78	0.78	0.77	0.77	0.77	0.77	0.77	0.77	0.77	0.76	0.76	0.76	0.76	0.76	0.75	0.75	0.75	0.75	0.75	3
4	1.04	1.03	1.03	1.03	1.03	1.03	1.03	1.02	1.02	1.02	1.02	1.01	1.01	1.01	1.01	1.00	1.00	1.00	1.00	4
5	1.29	1.29	1.29	1.29	1.29	1.28	1.28	1.28	1.28	1.27	1.27	1.27	1.27	1.26	1.26	1.25	1.25	1.25	1.25	5
6	1.55	1.55	1.55	1.55	1.55	1.54	1.54	1.53	1.53	1.53	1.53	1.52	1.52	1.51	1.51	1.51	1.51	1.50	1.50	6
7	1.81	1.81	1.81	1.80	1.80	1.80	1.80	1.79	1.79	1.79	1.78	1.77	1.77	1.77	1.76	1.76	1.76	1.75	1.75	7
8	2.06	2.06	2.06	2.06	2.06	2.06	2.05	2.04	2.04	2.04	2.03	2.03	2.02	2.02	2.02	2.01	2.01	2.00	2.00	8
9	2.31	2.31	2.32	2.32	2.31	2.31	2.30	2.30	2.30	2.29	2.28	2.28	2.28	2.27	2.27	2.26	2.26	2.25	2.25	9
10	2.57	2.57	2.58	2.58	2.57	2.57	2.56	2.56	2.55	2.55	2.54	2.54	2.53	2.53	2.52	2.51	2.51	2.50	2.50	10
11	2.83	2.83	2.83	2.83	2.83	2.82	2.82	2.81	2.81	2.80	2.80	2.79	2.78	2.78	2.77	2.76	2.76	2.75	2.75	11
12	3.08	3.08	3.08	3.08	3.08	3.07	3.07	3.06	3.06	3.05	3.05	3.04	3.04	3.03	3.02	3.01	3.01	3.00	3.00	12
13	3.34	3.34	3.34	3.34	3.34	3.33	3.33	3.32	3.32	3.31	3.30	3.30	3.29	3.28	3.28	3.27	3.26	3.26	3.26	13
14	3.59	3.59	3.59	3.59	3.59	3.58	3.58	3.58	3.57	3.56	3.56	3.55	3.54	3.53	3.52	3.52	3.51	3.51	3.51	14
15	3.84	3.84	3.83	3.83	3.83	3.83	3.82	3.82	3.82	3.82	3.81	3.81	3.79	3.78	3.78	3.77	3.76	3.76	3.76	15
16	4.09	4.09	4.09	4.09	4.09	4.09	4.08	4.08	4.07	4.07	4.07	4.06	4.05	4.04	4.03	4.02	4.01	4.01	4.01	16
17	4.34	4.34	4.33	4.33	4.33	4.33	4.33	4.32	4.32	4.32	4.31	4.30	4.30	4.29	4.28	4.28	4.27	4.26	4.26	17
18	4.59	4.59	4.58	4.58	4.58	4.58	4.58	4.58	4.57	4.57	4.57	4.56	4.55	4.54	4.54	4.53	4.52	4.51	4.51	18
19	4.84	4.84	4.84	4.84	4.84	4.84	4.84	4.84	4.84	4.83	4.83	4.82	4.81	4.80	4.79	4.78	4.77	4.76	4.76	19
20	5.09	5.09	5.09	5.09	5.09	5.09	5.09	5.09	5.09	5.08	5.08	5.07	5.06	5.05	5.04	5.03	5.02	5.01	5.01	20
21	5.34	5.34	5.33	5.33	5.33	5.33	5.33	5.33	5.33	5.32	5.32	5.32	5.31	5.30	5.29	5.28	5.27	5.26	5.26	21
22	5.59	5.59	5.59	5.59	5.59	5.59	5.59	5.59	5.59	5.59	5.58	5.58	5.57	5.56	5.54	5.53	5.52	5.51	5.51	22

\* Handbook of Sugar Analysis, 118.



TABLE 36—Continued

SCHMITZ'S TABLE (RECALCULATED) FOR SUCROSE FOR USE IN HORNE'S DRY LEAD METHOD AND IN POLARIZING UNDLUTED SOLUTIONS

Degrees Brix and Per Cents Sucrose																					
Pol. Reading	10.5	11.0	11.5	12.0	12.5	13.0	13.5	14.0	14.5	15.0	15.5	16.0	16.5	17.0	17.5	18.0	18.5	19.0	19.5	20.0	Pol. Reading
1	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.24	0.24	0.24	0.24	0.24	0.24	0.24	0.24	0.24	1
2	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.48	0.48	0.48	0.48	0.48	0.48	0.48	0.48	0.48	2
3	0.75	0.75	0.75	0.75	0.74	0.74	0.74	0.74	0.74	0.74	0.74	0.73	0.73	0.73	0.73	0.73	0.73	0.73	0.72	0.72	3
4	1.00	1.00	1.00	0.99	0.99	0.99	0.99	0.99	0.99	0.98	0.98	0.98	0.98	0.97	0.97	0.97	0.97	0.96	0.96	0.96	4
5	1.25	1.25	1.25	1.24	1.24	1.24	1.24	1.24	1.23	1.23	1.23	1.22	1.22	1.22	1.22	1.21	1.21	1.21	1.20	1.20	5
6	1.50	1.50	1.50	1.49	1.49	1.49	1.48	1.48	1.48	1.47	1.47	1.47	1.46	1.46	1.45	1.45	1.45	1.45	1.44	1.44	6
7	1.75	1.75	1.75	1.74	1.74	1.74	1.73	1.73	1.72	1.72	1.71	1.71	1.70	1.70	1.69	1.69	1.69	1.69	1.68	1.68	7
8	2.00	2.00	1.99	1.98	1.98	1.98	1.97	1.97	1.97	1.96	1.96	1.95	1.95	1.95	1.94	1.94	1.94	1.93	1.93	1.92	8
9	2.25	2.24	2.24	2.24	2.24	2.24	2.23	2.23	2.22	2.21	2.21	2.21	2.20	2.20	2.19	2.19	2.18	2.18	2.17	2.16	9
10	2.50	2.49	2.49	2.48	2.48	2.48	2.47	2.47	2.46	2.46	2.45	2.45	2.44	2.44	2.43	2.43	2.42	2.42	2.41	2.41	10
11	2.75	2.74	2.73	2.73	2.72	2.72	2.71	2.71	2.70	2.70	2.69	2.68	2.68	2.67	2.67	2.66	2.66	2.65	2.65	2.64	11
12	3.00	2.99	2.98	2.98	2.97	2.97	2.96	2.96	2.95	2.95	2.94	2.93	2.93	2.92	2.92	2.91	2.90	2.89	2.89	2.89	12
13	3.25	3.24	3.23	3.23	3.22	3.21	3.20	3.20	3.19	3.18	3.18	3.17	3.17	3.16	3.16	3.15	3.14	3.13	3.13	3.13	13
14	3.50	3.50	3.49	3.48	3.47	3.46	3.45	3.45	3.44	3.43	3.42	3.42	3.41	3.41	3.41	3.40	3.39	3.38	3.37	3.37	14
15	3.75	3.74	3.73	3.72	3.71	3.70	3.69	3.69	3.68	3.67	3.67	3.66	3.66	3.65	3.65	3.64	3.63	3.62	3.62	3.61	15
16	4.00	3.99	3.98	3.97	3.96	3.96	3.95	3.94	3.93	3.93	3.92	3.91	3.90	3.89	3.88	3.88	3.87	3.86	3.85	3.85	16
17	4.25	4.25	4.23	4.23	4.21	4.20	4.19	4.18	4.17	4.17	4.16	4.15	4.14	4.13	4.12	4.12	4.11	4.11	4.10	4.09	17
18	4.50	4.49	4.48	4.47	4.46	4.45	4.44	4.44	4.43	4.42	4.41	4.40	4.39	4.37	4.37	4.36	4.35	4.34	4.33	4.33	18
19	4.75	4.74	4.73	4.72	4.71	4.70	4.69	4.68	4.67	4.66	4.65	4.64	4.63	4.61	4.61	4.61	4.60	4.59	4.58	4.57	19
20	5.00	4.99	4.98	4.97	4.96	4.95	4.94	4.93	4.93	4.91	4.90	4.89	4.88	4.86	4.86	4.85	4.84	4.83	4.82	4.81	20
21	5.25	5.23	5.22	5.21	5.20	5.19	5.18	5.17	5.16	5.15	5.14	5.13	5.12	5.10	5.10	5.09	5.08	5.07	5.06	5.05	21
22	5.50	5.48	5.47	5.46	5.45	5.44	5.43	5.42	5.41	5.40	5.39	5.38	5.37	5.35	5.35	5.34	5.32	5.31	5.30	5.29	22
23	5.75	5.73	5.72	5.71	5.70	5.69	5.68	5.67	5.66	5.65	5.64	5.63	5.61	5.59	5.58	5.58	5.56	5.55	5.54	5.53	23
24	6.00	5.98	5.97	5.96	5.95	5.94	5.93	5.92	5.91	5.90	5.89	5.87	5.86	5.84	5.83	5.82	5.81	5.80	5.79	5.77	24
25	6.25	6.23	6.22	6.21	6.20	6.18	6.17	6.16	6.15	6.14	6.13	6.11	6.10	6.08	6.07	6.06	6.05	6.04	6.03	6.01	25

TABLE 36—Continued

SCHMIDT'S TABLE (RECALCULATED) FOR SUCROSE FOR USE IN HORNE'S DRY LEAD METHOD AND IN POLARIZING UNDLUTED SOLUTIONS

Degrees Brix and Per Cents Sucrose																					
Pol. Reading	10.5	11.0	11.5	12.0	12.5	13.0	13.5	14.0	14.5	15.0	15.5	16.0	16.5	17.0	17.5	18.0	18.5	19.0	19.5	20.0	Pol. Reading
26	6.50	6.49	6.48	6.46	6.44	6.43	6.42	6.41	6.40	6.39	6.38	6.36	6.34	6.33	6.32	6.30	6.29	6.28	6.27	6.26	26
27	6.76	6.74	6.72	6.70	6.69	6.68	6.67	6.65	6.64	6.63	6.62	6.60	6.59	6.57	6.56	6.55	6.54	6.52	6.51	6.50	27
28	7.00	6.98	6.97	6.95	6.94	6.93	6.93	6.90	6.89	6.87	6.86	6.85	6.84	6.82	6.81	6.79	6.78	6.77	6.75	6.74	28
29	7.25	7.23	7.22	7.20	7.19	7.17	7.16	7.15	7.14	7.12	7.11	7.09	7.08	7.06	7.05	7.03	7.02	7.01	6.99	6.98	29
30	7.50	7.48	7.47	7.45	7.44	7.43	7.41	7.40	7.39	7.37	7.35	7.34	7.32	7.31	7.29	7.27	7.26	7.25	7.23	7.22	30
31	7.75	7.73	7.71	7.70	7.68	7.66	7.65	7.64	7.63	7.61	7.60	7.58	7.57	7.55	7.53	7.51	7.50	7.49	7.47	7.46	31
32	8.00	7.98	7.96	7.95	7.93	7.91	7.90	7.89	7.88	7.86	7.84	7.82	7.81	7.79	7.78	7.76	7.75	7.73	7.71	7.70	32
33	8.25	8.23	8.21	8.20	8.18	8.16	8.15	8.13	8.12	8.10	8.09	8.07	8.05	8.03	8.01	8.00	7.99	7.97	7.95	7.94	33
34	8.50	8.48	8.47	8.45	8.43	8.41	8.40	8.38	8.37	8.35	8.34	8.32	8.30	8.28	8.26	8.24	8.23	8.22	8.20	8.18	34
35	8.75	8.73	8.71	8.69	8.68	8.66	8.65	8.63	8.61	8.59	8.58	8.56	8.54	8.52	8.50	8.48	8.47	8.46	8.44	8.42	35
36	9.00	8.98	8.96	8.94	8.92	8.91	8.90	8.88	8.86	8.84	8.82	8.80	8.78	8.76	8.75	8.73	8.71	8.70	8.68	8.66	36
37	9.25	9.23	9.21	9.19	9.17	9.16	9.14	9.12	9.10	9.08	9.07	9.05	9.03	9.01	8.99	8.97	8.95	8.94	8.92	8.90	37
38	9.50	9.48	9.46	9.44	9.42	9.40	9.39	9.37	9.35	9.33	9.31	9.29	9.27	9.26	9.24	9.21	9.20	9.18	9.16	9.14	38
39	9.75	9.73	9.71	9.69	9.67	9.65	9.64	9.62	9.60	9.58	9.56	9.53	9.51	9.50	9.48	9.46	9.44	9.43	9.40	9.38	39

Degrees Brix from 0.5 to 12.0			Degrees Brix from 12.0 to 20.0		
Tenths of the Polari- scope Reading		Per Cents Sucrose	Tenths of the Polari- scope Reading		Per Cents Sucrose
0.1		0.02	0.1		0.02
0.2		0.05	0.2		0.05
0.3		0.07	0.3		0.07
0.4		0.10	0.4		0.10
0.5			0.5		0.12
0.6		0.12	0.6		0.15
0.7		0.15	0.7		0.17
0.8		0.17	0.8		0.19
0.9		0.20	0.9		0.22
		0.22			0.22

TABLE 36—Continued

SCHMITZ'S TABLE (RECALCULATED) FOR SUCROSE FOR USE IN HORNE'S DRY LEAD METHOD AND IN POLARIZING UNDLUTED SOLUTIONS

Degrees Brix and Per Cents Sucrose																		
Pol. Reading	11.5	12.0	12.5	13.0	13.5	14.0	14.5	15.0	15.5	16.0	16.5	17.0	17.5	18.0	18.5	19.0	19.5	Pol. Reading
40	9.96	9.94	9.92	9.90	9.88	9.85	9.84	9.82	9.80	9.78	9.76	9.74	9.73	9.70	9.68	9.66	9.64	40
41	10.20	10.18	10.17	10.15	10.12	10.10	10.08	10.06	10.04	10.02	10.00	9.98	9.96	9.94	9.92	9.90	9.88	41
42	10.45	10.43	10.42	10.40	10.37	10.35	10.33	10.31	10.29	10.26	10.24	10.22	10.20	10.18	10.16	10.14	10.12	42
43	10.70	10.68	10.67	10.65	10.62	10.60	10.58	10.56	10.53	10.51	10.49	10.47	10.44	10.42	10.41	10.38	10.36	43
44	10.95	10.93	10.91	10.89	10.86	10.85	10.83	10.81	10.78	10.76	10.74	10.72	10.69	10.67	10.65	10.63	10.60	44
45	11.20	11.18	11.16	11.14	11.11	11.09	11.07	11.05	11.02	11.00	10.98	10.96	10.93	10.91	10.89	10.87	10.85	45
46	11.45	11.43	11.41	11.39	11.36	11.34	11.32	11.29	11.27	11.25	11.23	11.20	11.18	11.16	11.14	11.11	11.08	46
47	11.70	11.68	11.66	11.64	11.61	11.59	11.56	11.54	11.51	11.49	11.47	11.44	11.42	11.40	11.37	11.35	11.33	47
48	11.95	11.93	11.91	11.89	11.86	11.84	11.81	11.79	11.76	11.74	11.72	11.69	11.66	11.64	11.62	11.59	11.56	48
49	12.20	12.18	12.15	12.13	12.10	12.08	12.05	12.03	12.00	11.98	11.96	11.93	11.90	11.88	11.86	11.83	11.80	49
50	12.45	12.43	12.41	12.38	12.35	12.33	12.30	12.28	12.25	12.23	12.20	12.18	12.15	12.13	12.10	12.08	12.05	50
51	12.70	12.68	12.66	12.63	12.60	12.57	12.54	12.52	12.49	12.47	12.44	12.42	12.39	12.37	12.34	12.32	12.29	51
52	12.95	12.93	12.91	12.88	12.85	12.82	12.79	12.77	12.74	12.71	12.69	12.67	12.63	12.62	12.58	12.56	12.53	52
53	13.20	13.18	13.16	13.13	13.10	13.07	13.04	13.01	12.99	12.96	12.93	12.91	12.87	12.86	12.83	12.80	12.77	53
54	13.45	13.43	13.41	13.38	13.35	13.32	13.29	13.26	13.24	13.20	13.18	13.15	13.12	13.11	13.07	13.04	13.01	54
55	13.70	13.68	13.66	13.63	13.60	13.57	13.54	13.51	13.48	13.44	13.42	13.39	13.36	13.34	13.31	13.28	13.25	55
56	13.95	13.93	13.91	13.88	13.85	13.82	13.79	13.75	13.73	13.69	13.66	13.64	13.61	13.58	13.55	13.52	13.50	56
57	14.20	14.18	14.16	14.13	14.10	14.07	14.04	14.01	13.99	13.95	13.93	13.90	13.88	13.85	13.82	13.79	13.76	57
58	14.45	14.43	14.41	14.38	14.35	14.32	14.29	14.26	14.24	14.21	14.18	14.15	14.12	14.09	14.04	14.01	13.98	58
59	14.70	14.68	14.66	14.63	14.60	14.57	14.54	14.51	14.48	14.45	14.42	14.39	14.36	14.33	14.28	14.25	14.22	59
60	14.95	14.93	14.91	14.88	14.85	14.82	14.79	14.76	14.73	14.70	14.68	14.65	14.62	14.59	14.56	14.53	14.50	60
61	15.20	15.18	15.16	15.13	15.10	15.07	15.04	15.01	14.99	14.96	14.93	14.90	14.87	14.84	14.81	14.78	14.75	61
62	15.45	15.43	15.41	15.38	15.35	15.32	15.29	15.26	15.24	15.20	15.18	15.15	15.12	15.09	15.04	15.01	14.98	62
63	15.70	15.68	15.66	15.63	15.60	15.57	15.54	15.51	15.48	15.45	15.42	15.39	15.36	15.33	15.29	15.26	15.23	63
64	15.95	15.93	15.91	15.88	15.85	15.82	15.79	15.75	15.73	15.69	15.66	15.64	15.61	15.58	15.54	15.51	15.48	64
65	16.20	16.18	16.16	16.13	16.10	16.07	16.04	16.01	15.99	15.95	15.93	15.90	15.87	15.84	15.80	15.77	15.74	65
66	16.45	16.43	16.41	16.38	16.35	16.32	16.29	16.26	16.24	16.20	16.18	16.15	16.12	16.09	16.04	16.01	15.98	66
67	16.70	16.68	16.66	16.63	16.60	16.57	16.54	16.51	16.48	16.45	16.42	16.39	16.36	16.33	16.29	16.26	16.23	67
68	16.95	16.93	16.91	16.88	16.85	16.82	16.79	16.75	16.73	16.69	16.66	16.64	16.61	16.58	16.54	16.51	16.48	68
69	17.20	17.18	17.16	17.13	17.10	17.07	17.04	17.01	16.99	16.95	16.93	16.90	16.87	16.84	16.80	16.77	16.74	69
70	17.45	17.43	17.41	17.38	17.35	17.32	17.29	17.26	17.24	17.20	17.18	17.15	17.12	17.09	17.04	17.01	16.98	70
71	17.70	17.68	17.66	17.63	17.60	17.57	17.54	17.51	17.48	17.45	17.42	17.39	17.36	17.33	17.29	17.26	17.23	71

Degrees Brix from 11.5 to 22.5		
Tenths of the Polariscope Reading	Per Cents Sucrose	
	Pol. R.	Brix, etc.
0.1	72	17.39
0.2	73	17.63
0.3	74	17.87
0.4	75	18.11
0.5	76	18.35
0.6		18.59
0.7		18.83
0.8		19.07
0.9		19.31

TABLE 36—Continued

SCHMITZ'S TABLE (RECALCULATED) FOR SUCROSE FOR USE IN HORNE'S DRY LEAD METHOD AND IN POLARIZING UNDILUTED SOLUTIONS

Pol. Reading	Degrees Brix and Per Cents Sucrose									Pol. Reading
	20.0	20.5	21.0	21.5	22.0	22.5	23.0	23.5	24.0	
40	9.62	9.60	9.58	9.56	9.54	9.52	9.50	9.48	9.46	40
41	9.86	9.84	9.82	9.80	9.78	9.76	9.74	9.72	9.70	41
42	10.10	10.08	10.06	10.04	10.02	10.00	9.98	9.96	9.94	42
43	10.34	10.32	10.30	10.28	10.26	10.24	10.22	10.20	10.18	43
44	10.58	10.56	10.54	10.52	10.49	10.47	10.46	10.44	10.41	44
45	10.82	10.80	10.78	10.76	10.73	10.71	10.69	10.67	10.64	45
46	11.06	11.04	11.02	11.00	10.97	10.95	10.93	10.90	10.88	46
47	11.30	11.28	11.26	11.24	11.21	11.19	11.17	11.14	11.12	47
48	11.54	11.52	11.50	11.48	11.45	11.43	11.41	11.38	11.36	48
49	11.78	11.76	11.74	11.72	11.69	11.67	11.65	11.62	11.60	49
50	12.03	12.00	11.98	11.95	11.93	11.90	11.88	11.85	11.83	50
51	12.27	12.24	12.22	12.19	12.17	12.14	12.12	12.09	12.07	51
52	12.51	12.48	12.46	12.43	12.41	12.38	12.35	12.33	12.30	52
53	12.75	12.72	12.70	12.67	12.65	12.62	12.59	12.57	12.53	53
54	12.98	12.96	12.94	12.91	12.89	12.86	12.83	12.81	12.77	54
55	13.22	13.20	13.17	13.15	13.12	13.09	13.06	13.04	13.01	55
56	13.47	13.44	13.41	13.39	13.36	13.33	13.30	13.28	13.24	56
57	13.71	13.68	13.65	13.63	13.60	13.57	13.54	13.52	13.48	57
58	13.95	13.92	13.89	13.87	13.84	13.81	13.77	13.75	13.72	58
59	14.19	14.16	14.13	14.11	14.08	14.05	14.01	13.98	13.96	59
60	14.43	14.40	14.37	14.34	14.31	14.28	14.25	14.22	14.19	60
61	14.67	14.64	14.61	14.58	14.55	14.52	14.49	14.46	14.42	61
62	14.91	14.88	14.85	14.82	14.79	14.76	14.73	14.69	14.66	62
63	15.15	15.12	15.09	15.06	15.03	14.99	14.96	14.93	14.90	63
64	15.39	15.36	15.33	15.30	15.27	15.24	15.20	15.17	15.14	64
65	15.63	15.60	15.57	15.54	15.51	15.48	15.44	15.41	15.38	65
66	15.87	15.84	15.81	15.78	15.74	15.71	15.68	15.64	15.61	66
67	16.11	16.08	16.05	16.02	15.98	15.95	15.92	15.88	15.85	67
68	16.35	16.32	16.29	16.26	16.22	16.19	16.16	16.12	16.09	68
69	16.59	16.56	16.53	16.50	16.46	16.43	16.39	16.35	16.33	69
70	16.84	16.80	16.77	16.73	16.70	16.66	16.63	16.59	16.56	70
71	17.08	17.04	17.00	16.97	16.93	16.89	16.86	16.83	16.79	71
72	17.32	17.28	17.24	17.21	17.17	17.13	17.10	17.07	17.03	72
73	17.56	17.52	17.48	17.45	17.41	17.37	17.34	17.31	17.27	73
74	17.80	17.76	17.72	17.69	17.65	17.61	17.58	17.55	17.50	74
75	18.04	18.00	17.96	17.93	17.89	17.85	17.81	17.78	17.74	75
76	18.28	18.24	18.20	18.17	18.13	18.09	18.05	18.02	17.98	76
77	18.52	18.48	18.44	18.41	18.37	18.33	18.29	18.26	18.22	77
78	18.76	18.72	18.68	18.65	18.61	18.57	18.53	18.49	18.45	78
79	19.00	18.96	18.92	18.89	18.85	18.81	18.77	18.73	18.69	79
80	19.24	19.20	19.16	19.12	19.08	19.04	19.00	18.96	18.92	80
81	.....	19.44	19.40	19.36	19.32	19.28	19.24	19.20	19.16	81
82	.....	19.68	19.64	19.60	19.56	19.52	19.48	19.44	19.40	82
83	.....	.....	19.88	19.84	19.80	19.76	19.72	19.68	19.64	83
84	.....	.....	20.12	20.08	20.04	19.99	19.95	19.92	19.88	84
85	.....	.....	.....	20.32	20.28	20.23	20.19	20.15	20.10	85

Degrees Brix from 23 to 24

Tenths of the Polariscope Reading	Per Cents Sucrose	Tenths of the Polariscope Reading	Per Cents Sucrose
0.1	0.02	0.5	0.12
0.2	0.05	0.6	0.14
0.3	0.07	0.7	0.17
0.4	0.09	0.8	0.19
		0.9	0.21

TABLE 37

AVAILABLE SUGAR TABLES \*

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Values of the factor  $1.4 - \frac{\text{Coefficient purity}}{\text{Coefficient purity}} \times 100$ , for coefficients of purity ranging from 77 to 93, advancing by tenths. See page 359 for suggestions relative to the use of these tables.

(A) SUCROSE

Purity Coefficient	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
77	88.05	88.12	88.19	88.25	88.32	88.39	88.45	88.52	88.59	88.65
78	88.72	88.78	88.85	88.92	88.98	89.04	89.11	89.18	89.24	89.31
79	89.37	89.43	89.50	89.56	89.63	89.69	89.75	89.81	89.88	89.94
80	90.00	90.07	90.13	90.19	90.25	90.32	90.38	90.44	90.50	90.56
81	90.62	90.68	90.74	90.80	90.86	90.92	90.98	91.04	91.10	91.16
82	91.22	91.28	91.34	91.39	91.45	91.51	91.57	91.63	91.69	91.75
83	91.81	91.87	91.93	91.99	92.04	92.10	92.15	92.21	92.27	92.32
84	92.38	92.44	92.50	92.55	92.61	92.67	92.72	92.78	92.84	92.89
85	92.94	93.00	93.06	93.11	93.16	93.22	93.28	93.33	93.38	93.43
86	93.49	93.54	93.59	93.64	93.70	93.75	93.80	93.85	93.91	93.97
87	94.02	94.07	94.12	94.18	94.23	94.28	94.34	94.39	94.44	94.49
88	94.55	94.60	94.65	94.70	94.76	94.81	94.86	94.91	94.95	95.01
89	95.06	95.11	95.16	95.21	95.26	95.31	95.36	95.41	95.46	95.51
90	95.56	95.61	95.65	95.70	95.75	95.80	95.85	95.90	95.95	96.00
91	96.04	96.09	96.14	96.18	96.23	96.28	96.33	96.37	96.42	96.47
92	96.52	96.57	96.62	96.67	96.71	96.75	96.80	96.85	96.90	96.95
93	96.99	97.04	97.08	97.13	97.17	97.22	97.27	97.31	97.35	97.40

\* Methods of Chemical Control, H. C. Prinsen Geerligs. (Section "A" only.)

TABLE 38—Continued

FOR THE DETERMINATION OF COEFFICIENTS OF PURITY.—(G. KOTTMANN)

Per Cent Sucrose	Per Cent of Non-Sucrose = Degree Brix Minus Per Cent Sucrose									Per Cent Sucrose
	1.9	2.0	2.1	2.2	2.3	2.4	2.5	2.6	2.7	
8.0	80.8	80.0	79.2	78.4	77.7	76.9	76.2	75.5	74.8	8.0
.2	81.2	80.4	79.6	78.8	78.1	77.4	76.6	75.9	75.2	.2
.4	81.5	80.8	80.0	79.2	78.5	77.8	77.1	76.4	75.7	.4
.6	81.9	81.1	80.4	79.6	78.9	78.2	77.5	76.8	76.1	.6
.8	82.2	81.5	80.7	80.0	79.3	78.6	77.9	77.2	76.5	.8
9.0	82.6	81.8	81.1	80.4	79.6	78.9	78.3	77.6	76.9	9.0
.2	82.9	82.1	81.4	80.7	80.0	79.3	78.6	77.9	77.3	.2
.4	83.2	82.5	81.7	81.0	80.3	79.7	79.0	78.3	77.7	.4
.6	83.5	82.8	82.1	81.4	80.7	80.0	79.3	78.7	78.0	.6
.8	83.8	83.1	82.4	81.7	81.0	80.3	79.7	79.0	78.4	.8
10.0	84.0	83.3	82.6	82.0	81.3	80.6	80.0	79.4	78.7	10.0
.2	84.3	83.6	82.9	82.3	81.6	81.0	80.3	79.7	79.1	.2
.4	84.6	83.9	83.2	82.5	81.9	81.2	80.6	80.0	79.4	.4
.6	84.8	84.1	83.5	82.8	82.2	81.5	80.9	80.3	79.7	.6
.8	85.0	84.4	83.7	83.1	82.4	81.8	81.2	80.6	80.0	.8
11.0	85.3	84.6	84.0	83.3	82.7	82.1	81.5	80.9	80.3	11.0
.2	85.5	84.8	84.2	83.6	83.0	82.4	81.8	81.2	80.6	.2
.4	85.7	85.1	84.4	83.8	83.2	82.6	82.0	81.4	80.9	.4
.6	85.9	85.3	84.7	84.1	83.5	82.9	82.3	81.7	81.1	.6
.8	86.1	85.5	84.9	84.3	83.7	83.1	82.5	81.9	81.4	.8
12.0	86.3	85.7	85.1	84.5	83.9	83.3	82.8	82.2	81.6	12.0
.2	86.5	85.9	85.3	84.7	84.1	83.6	83.0	82.4	81.9	.2
.4	86.7	86.1	85.5	84.9	84.4	83.8	83.2	82.7	82.1	.4
.6	86.9	86.3	85.7	85.1	84.6	84.0	83.4	82.9	82.4	.6
.8	87.1	86.5	85.9	85.3	84.8	84.2	83.7	83.1	82.6	.8
13.0	87.2	86.7	86.1	85.5	85.0	84.4	83.9	83.3	82.8	13.0
.2	87.4	86.8	86.3	85.7	85.2	84.6	84.1	83.5	83.0	.2
.4	87.6	87.0	86.5	85.9	85.4	84.8	84.3	83.7	83.2	.4
.6	87.7	87.2	86.6	86.1	85.5	85.0	84.5	83.9	83.4	.6
.8	87.9	87.3	86.8	86.3	85.7	85.2	84.7	84.1	83.6	.8
14.0	88.1	87.5	87.0	86.4	85.9	85.4	84.8	84.3	83.8	14.0
.2	88.2	87.7	87.1	86.6	86.1	85.5	85.0	84.5	84.0	.2
.4	88.3	87.8	87.3	86.7	86.2	85.7	85.2	84.7	84.2	.4
.6	88.5	88.0	87.4	86.9	86.4	85.9	85.4	84.9	84.4	.6
.8	88.6	88.1	87.6	87.1	86.5	86.0	85.5	85.1	84.6	.8
15.0	88.8	88.2	87.7	87.2	86.7	86.2	85.7	85.2	84.7	15.0
.2	88.9	88.4	87.9	87.4	86.9	86.4	85.9	85.4	84.9	.2
.4	89.0	88.5	88.0	87.5	87.0	86.5	86.0	85.6	85.1	.4
.6	89.1	88.6	88.1	87.6	87.2	86.7	86.2	85.7	85.2	.6
.8	89.3	88.8	88.3	87.8	87.3	86.8	86.3	85.9	85.4	.8
16.0	89.4	88.9	88.4	87.9	87.4	87.0	86.5	86.0	85.6	16.0
.2	89.5	89.0	88.5	88.0	87.6	87.1	86.6	86.2	85.7	.2
.4	89.6	89.1	88.6	88.2	87.7	87.2	86.8	86.3	85.9	.4
.6	89.7	89.2	88.8	88.3	87.8	87.4	86.9	86.5	86.0	.6
.8	89.8	89.4	88.9	88.4	88.0	87.5	87.0	86.6	86.2	.8
17.0	89.9	89.5	89.0	88.5	88.1	87.6	87.2	86.7	86.3	17.0



TABLE 38—Continued

FOR THE DETERMINATION OF COEFFICIENTS OF PURITY.—(G. KOTTMANN)

Per Cent Sucrose	Per Cent of Non-Sucrose = Degree Brix Minus Per Cent Sucrose									Per Cent Sucrose
	2.8	2.9	3.0	3.1	3.2	3.3	3.4	3.5	3.6	
8.0	74.1	73.4	72.7	72.1	71.4	70.8	70.2	69.6	69.0	8.0
.2	74.5	73.9	73.2	72.6	71.9	71.3	70.7	70.1	69.5	.2
.4	75.0	74.3	73.7	73.0	72.4	71.8	71.2	70.6	70.0	.4
.6	75.4	74.8	74.1	73.5	72.9	72.3	71.7	71.1	70.5	.6
.8	75.9	75.2	74.6	73.9	73.3	72.7	72.1	71.5	71.0	.8
9.0	76.3	75.6	75.0	74.4	73.8	73.2	72.6	72.0	71.4	9.0
.2	76.7	76.0	75.4	74.8	74.2	73.6	73.0	72.4	71.9	.2
.4	77.0	76.4	75.8	75.2	74.6	74.0	73.4	72.9	72.3	.4
.6	77.4	76.8	76.2	75.6	75.0	74.4	73.8	73.3	72.7	.6
.8	77.8	77.2	76.6	76.0	75.4	74.8	74.2	73.7	73.1	.8
10.0	78.1	77.5	76.9	76.3	75.8	75.2	74.6	74.1	73.5	10.0
.2	78.5	77.9	77.3	76.7	76.1	75.6	75.0	74.5	73.9	.2
.4	78.8	78.2	77.6	77.0	76.5	75.9	75.4	74.8	74.3	.4
.6	79.1	78.5	77.9	77.4	76.8	76.3	75.7	75.2	74.6	.6
.8	79.4	78.8	78.3	77.7	77.1	76.6	76.1	75.5	75.0	.8
11.0	79.7	79.1	78.6	78.0	77.5	76.9	76.4	75.9	75.3	11.0
.2	80.0	79.4	78.9	78.3	77.8	77.2	76.7	76.2	75.7	.2
.4	80.3	79.7	79.2	78.6	78.1	77.6	77.0	76.5	76.0	.4
.6	80.6	80.0	79.4	78.9	78.4	77.9	77.3	76.8	76.3	.6
.8	80.8	80.3	79.7	79.2	78.7	78.1	77.6	77.1	76.6	.8
12.0	81.1	80.5	80.0	79.5	78.9	78.4	77.9	77.4	76.9	12.0
.2	81.3	80.8	80.3	79.7	79.2	78.7	78.2	77.7	77.2	.2
.4	81.6	81.0	80.5	80.0	79.5	79.0	78.5	78.0	77.5	.4
.6	81.8	81.3	80.8	80.3	79.7	79.2	78.8	78.3	77.8	.6
.8	82.1	81.5	81.0	80.5	80.0	79.5	79.0	78.5	78.0	.8
13.0	82.3	81.8	81.2	80.7	80.2	79.8	79.3	78.8	78.3	13.0
.2	82.5	82.0	81.5	81.0	80.5	80.0	79.5	79.0	78.6	.2
.4	82.7	82.2	81.7	81.2	80.7	80.2	79.8	79.3	78.8	.4
.6	82.9	82.4	81.9	81.4	81.0	80.5	80.0	79.5	79.1	.6
.8	83.1	82.6	82.1	81.7	81.2	80.7	80.2	79.8	79.3	.8
14.0	83.3	82.8	82.3	81.9	81.4	80.9	80.5	80.0	79.5	14.0
.2	83.5	83.0	82.5	82.1	81.6	81.1	80.7	80.2	79.8	.2
.4	83.7	83.2	82.7	82.3	81.8	81.4	80.9	80.4	80.0	.4
.6	83.9	83.4	82.9	82.5	82.0	81.6	81.1	80.7	80.2	.6
.8	84.1	83.6	83.1	82.7	82.2	81.8	81.3	80.9	80.4	.8
15.0	84.3	83.8	83.3	82.9	82.4	82.0	81.5	81.1	80.6	15.0
.2	84.4	84.0	83.5	83.1	82.6	82.2	81.7	81.3	80.8	.2
.4	84.6	84.2	83.7	83.2	82.8	82.4	81.9	81.5	81.0	.4
.6	84.8	84.3	83.9	83.4	83.0	82.5	82.1	81.7	81.2	.6
.8	84.9	84.5	84.0	83.6	83.2	82.7	82.3	81.9	81.4	.8
16.0	85.1	84.7	84.2	83.8	83.3	82.9	82.5	82.0	81.6	16.0
.2	85.3	84.8	84.4	83.9	83.5	83.1	82.7	82.2	81.8	.2
.4	85.4	84.9	84.5	84.1	83.7	83.2	82.8	82.4	82.0	.4
.6	85.6	85.1	84.7	84.3	83.8	83.4	83.0	82.6	82.2	.6
.8	85.7	85.3	84.8	84.4	84.0	83.6	83.2	82.8	82.4	.8
17.0	85.9	85.4	85.0	84.6	84.2	83.7	83.3	82.9	82.5	17.0

TABLE 38—Continued

FOR THE DETERMINATION OF COEFFICIENTS OF PURITY—(G. KOTTMANN).

Per Cent Sucrose	Per Cent of Non-Sucrose = Degree Brix Minus Per Cent Sucrose									Per Cent Sucrose
	3.7	3.8	3.9	4.0	4.1	4.2	4.3	4.4	4.5	
8.0	68.4	67.8	67.2	66.7	66.1	65.6	65.0	64.5	64.0	8.0
.2	68.9	68.3	67.8	67.2	66.7	66.1	65.6	65.1	64.6	.2
.4	69.4	68.8	68.3	67.7	67.2	66.7	66.1	65.6	65.1	.4
.6	69.9	69.3	68.8	68.3	67.7	67.2	66.7	66.2	65.6	.6
.8	70.4	69.8	69.3	68.8	68.2	67.7	67.2	66.7	66.2	.8
9.0	70.9	70.3	69.8	69.2	68.7	68.2	67.7	67.2	66.7	9.0
.2	71.3	70.8	70.2	69.7	69.2	68.7	68.1	67.6	67.2	.2
.4	71.8	71.2	70.7	70.1	69.6	69.1	68.6	68.1	67.6	.4
.6	72.2	71.6	71.1	70.6	70.1	69.6	69.1	68.6	68.1	.6
.8	72.6	72.1	71.5	71.0	70.5	70.0	69.5	69.0	68.5	.8
10.0	73.0	72.5	71.9	71.4	70.9	70.4	69.9	69.4	69.0	10.0
.2	73.4	72.9	72.3	71.8	71.3	70.8	70.3	69.9	69.4	.2
.4	73.8	73.2	72.7	72.2	71.7	71.2	70.7	70.3	69.8	.4
.6	74.1	73.6	73.1	72.6	72.1	71.6	71.1	70.7	70.2	.6
.8	74.5	74.0	73.5	73.0	72.5	72.0	71.5	71.1	70.6	.8
11.0	74.8	74.3	73.8	73.3	72.8	72.4	71.9	71.4	71.0	11.0
.2	75.2	74.7	74.2	73.7	73.2	72.7	72.3	71.8	71.3	.2
.4	75.5	75.0	74.5	74.0	73.5	73.1	72.6	72.2	71.7	.4
.6	75.8	75.3	74.8	74.4	73.9	73.4	73.0	72.5	72.0	.6
.8	76.1	75.6	75.2	74.7	74.2	73.8	73.3	72.8	72.4	.8
12.0	76.4	75.9	75.5	75.0	74.5	74.1	73.6	73.2	72.7	12.0
.2	76.7	76.2	75.8	75.3	74.8	74.4	73.9	73.5	73.1	.2
.4	77.0	76.5	76.1	75.6	75.2	74.7	74.3	73.8	73.4	.4
.6	77.3	76.8	76.4	75.9	75.4	75.0	74.6	74.1	73.7	.6
.8	77.6	77.1	76.6	76.2	75.7	75.3	74.9	74.4	74.0	.8
13.0	77.8	77.4	76.9	76.5	76.0	75.6	75.1	74.7	74.3	13.0
.2	78.1	77.6	77.2	76.7	76.3	75.9	75.4	75.0	74.6	.2
.4	78.4	77.9	77.5	77.0	76.6	76.1	75.7	75.3	74.9	.4
.6	78.6	78.2	77.7	77.3	76.8	76.4	76.0	75.6	75.1	.6
.8	78.9	78.4	78.0	77.5	77.1	76.7	76.2	75.8	75.4	.8
14.0	79.1	78.7	78.2	77.8	77.3	76.9	76.5	76.1	75.7	14.0
.2	79.3	78.9	78.5	78.0	77.6	77.2	76.8	76.3	75.9	.2
.4	79.6	79.1	78.7	78.3	77.8	77.4	77.0	76.6	76.2	.4
.6	79.8	79.3	78.9	78.5	78.1	77.6	77.2	76.8	76.4	.6
.8	80.0	79.6	79.1	78.7	78.3	77.9	77.5	77.1	76.7	.8
15.0	80.2	79.8	79.4	78.9	78.5	78.1	77.7	77.3	76.9	15.0
.2	80.4	80.0	79.6	79.2	78.8	78.4	77.9	77.6	77.2	.2
.4	80.6	80.2	79.8	79.4	79.0	78.6	78.2	77.8	77.4	.4
.6	80.8	80.4	80.0	79.6	79.2	78.8	78.4	78.0	77.6	.6
.8	81.0	80.6	80.2	79.8	79.4	79.0	78.6	78.2	77.8	.8
16.0	81.2	80.8	80.4	80.0	79.6	79.2	78.8	78.4	78.0	16.0
.2	81.4	81.0	80.6	80.2	79.8	79.4	79.0	78.6	78.3	.2
.4	81.6	81.2	80.8	80.4	80.0	79.6	79.2	78.8	78.5	.4
.6	81.8	81.4	81.0	80.6	80.2	79.8	79.4	79.0	78.7	.6
.8	82.0	81.6	81.2	80.8	80.4	80.0	79.6	79.2	78.9	.8
17.0	82.1	81.7	81.3	81.0	80.6	80.2	79.8	79.4	79.1	17.0

EXPANDED HORNE'S TABLE FOR THE CALCULATION OF  
COEFFICIENTS OF PURITY

(Explanatory)

The fifth edition of this book (1915) included a table by Dr. W. D. Horne for use in calculating coefficients of purity with his dry lead defecation. The range of the table was from 15° to 16° Brix, which limited its usefulness. This table has been expanded to include all degrees Brix from 12 to 20. The table will prove useful in vacuum-pan and crystallizer control work and in refinery routine control. (See page 326 for method of making purities.) In the table on the pages following, opposite the degree Brix corrected to 20° C. and under the direct polariscope reading of the solution (after clarification with Horne's dry lead) will be found the coefficient of purity.

The table has been calculated using E. W. Rice's modification of Casamajor's formula for "water purities." The Rice formula is for densities at 20° C., true cubic centimeters (ml), and a normal weight of 26.0 grams, whereas Casamajor's was for the older units at 17½° C. and 26.048 normal weights. The formula according to Rice is

$$\text{Factor} = \frac{26 \times 100}{99.718 \times \text{sp. gr.} \times \text{Brix}}$$

The direct polariscope reading of the solution times the factor corresponding to the Brix gives the coefficient of purity as shown in the expanded Horne's table. The factors according to Rice's formula follow:

## PURITY FACTORS FOR USE WITH DRY LEAD DEFECATION.—(E. W. RICE) \*

°Bx.	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
0	.....	260.6336	130.2660	86.8102	65.0829	52.0461	43.3549	37.1469	32.4909	28.8695
1	25.9725	23.6022	21.6269	19.9556	18.5230	17.2814	16.1951	15.2365	14.3845	13.6221
2	12.9360	12.3152	11.7508	11.2355	10.7632	10.3286	9.9275	9.5561	9.2113	8.8901
3	8.5905	8.3102	8.0473	7.8004	7.5680	7.3490	7.1420	6.9463	6.7608	6.5849
4	6.4178	6.2588	6.1074	5.9630	5.8253	5.6936	5.5676	5.4470	5.3314	5.2208
5	5.1142	5.0120	4.9136	4.8191	4.7280	4.6402	4.5555	4.4738	4.3950	4.3188
6	4.2451	4.1739	4.1050	4.0382	3.9755	3.9109	3.8501	3.7912	3.7339	3.6784
7	3.6244	3.5720	3.5209	3.4713	3.4231	3.3761	3.3304	3.2858	3.2424	3.2001
8	3.1539	3.1186	3.0794	3.0411	3.0037	2.9671	2.9315	2.8966	2.8626	2.8293
9	2.7968	2.7649	2.7338	2.7033	2.6735	2.6443	2.6157	2.5877	2.5603	2.5334
10	2.5071	2.4813	2.4560	2.4312	2.4068	2.3830	2.3596	2.3366	2.3140	2.2919
11	2.2701	2.2488	2.2278	2.2072	2.1870	2.1671	2.1475	2.1283	2.1095	2.0909
12	2.0727	2.0547	2.0370	2.0197	2.0026	1.9858	1.9692	1.9529	1.9369	1.9211
13	1.9056	1.8903	1.8752	1.8603	1.8457	1.8313	1.8171	1.8031	1.7893	1.7757
14	1.7623	1.7491	1.7361	1.7233	1.7106	1.6981	1.6858	1.6737	1.6617	1.6499
15	1.6382	1.6267	1.6154	1.6042	1.5931	1.5822	1.5714	1.5608	1.5503	1.5399
16	1.5296	1.5195	1.5095	1.4997	1.4899	1.4803	1.4708	1.4614	1.4521	1.4429
17	1.4338	1.4249	1.4160	1.4072	1.3986	1.3900	1.3816	1.3732	1.3649	1.3567
18	1.3487	1.3407	1.3328	1.3249	1.3172	1.3095	1.3020	1.2945	1.2871	1.2797
19	1.2725	1.2653	1.2582	1.2511	1.2442	1.2373	1.2305	1.2237	1.2171	1.2104
20	1.2039	1.1974	1.1910	1.1846	1.1784	1.1721	1.1660	1.1598	1.1538	1.1478
21	1.1419	1.1360	1.1302	1.1244	1.1187	1.1130	1.1074	1.1018	1.0963	1.0909
22	1.0855	1.0801	1.0748	1.0696	1.0643	1.0592	1.0540	1.0490	1.0439	1.0389
23	1.0340	1.0291	1.0242	1.0194	1.0146	1.0099	1.0052	1.0005	.9959	.9913
24	.9868	.9823	.9778	.9734	.9690	.9647	.9603	.9560	.9518	.9476
25	.9434									

\* "Facts about Sugar," October 29, 1927

TABLE 39  
EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Poi. =	15.0	15.2	15.4	15.6	15.8	16.0	16.2	16.4	16.6	16.8	17.0	17.2	17.4	17.6	17.8	18.0	18.2	18.4	18.6	18.8	19.0	19.2	19.4	19.6	19.8
12.0	31.1	31.1	31.9	32.2	32.7	33.3	33.6	34.0	34.4	34.8	35.2	35.7	36.1	36.5	36.9	37.3	37.7	38.1	38.5	38.9	39.3	39.7	40.1	40.5	40.9
1	30.8	31.1	32.1	33.2	34.2	35.2	36.2	37.3	38.4	39.4	40.5	41.5	42.5	43.5	44.5	45.5	46.5	47.5	48.5	49.5	50.5	51.5	52.5	53.5	54.5
2	30.6	31.0	32.1	33.1	34.1	35.1	36.1	37.1	38.1	39.1	40.1	41.1	42.1	43.1	44.1	45.1	46.1	47.1	48.1	49.1	50.1	51.1	52.1	53.1	54.1
3	30.3	30.7	31.8	32.8	33.8	34.8	35.8	36.8	37.8	38.8	39.8	40.8	41.8	42.8	43.8	44.8	45.8	46.8	47.8	48.8	49.8	50.8	51.8	52.8	53.8
4	30.0	30.4	31.5	32.5	33.5	34.5	35.5	36.5	37.5	38.5	39.5	40.5	41.5	42.5	43.5	44.5	45.5	46.5	47.5	48.5	49.5	50.5	51.5	52.5	53.5
5	29.8	30.2	31.3	32.3	33.3	34.3	35.3	36.3	37.3	38.3	39.3	40.3	41.3	42.3	43.3	44.3	45.3	46.3	47.3	48.3	49.3	50.3	51.3	52.3	53.3
6	29.5	29.9	31.0	32.0	33.0	34.0	35.0	36.0	37.0	38.0	39.0	40.0	41.0	42.0	43.0	44.0	45.0	46.0	47.0	48.0	49.0	50.0	51.0	52.0	53.0
7	29.3	29.7	30.8	31.8	32.8	33.8	34.8	35.8	36.8	37.8	38.8	39.8	40.8	41.8	42.8	43.8	44.8	45.8	46.8	47.8	48.8	49.8	50.8	51.8	52.8
8	29.1	29.5	30.6	31.6	32.6	33.6	34.6	35.6	36.6	37.6	38.6	39.6	40.6	41.6	42.6	43.6	44.6	45.6	46.6	47.6	48.6	49.6	50.6	51.6	52.6
9	28.8	29.2	30.3	31.3	32.3	33.3	34.3	35.3	36.3	37.3	38.3	39.3	40.3	41.3	42.3	43.3	44.3	45.3	46.3	47.3	48.3	49.3	50.3	51.3	52.3
Poi. =	20.0	20.2	20.4	20.6	20.8	21.0	21.2	21.4	21.6	21.8	22.0	22.2	22.4	22.6	22.8	23.0	23.2	23.4	23.6	23.8	24.0	24.2	24.4	24.6	24.8
12.0	41.5	41.5	42.3	43.2	44.1	45.0	45.9	46.8	47.7	48.6	49.5	50.4	51.3	52.2	53.1	54.0	54.9	55.8	56.7	57.6	58.5	59.4	60.3	61.2	62.1
1	41.1	41.5	42.3	43.2	44.1	45.0	45.9	46.8	47.7	48.6	49.5	50.4	51.3	52.2	53.1	54.0	54.9	55.8	56.7	57.6	58.5	59.4	60.3	61.2	62.1
2	40.7	41.1	41.9	42.8	43.7	44.6	45.5	46.4	47.3	48.2	49.1	50.0	50.9	51.8	52.7	53.6	54.5	55.4	56.3	57.2	58.1	59.0	59.9	60.8	61.7
3	40.4	40.8	41.6	42.4	43.2	44.0	44.8	45.6	46.4	47.2	48.0	48.8	49.6	50.4	51.2	52.0	52.8	53.6	54.4	55.2	56.0	56.8	57.6	58.4	59.2
4	40.1	40.5	41.3	42.1	42.9	43.7	44.5	45.3	46.1	46.9	47.7	48.5	49.3	50.1	50.9	51.7	52.5	53.3	54.1	54.9	55.7	56.5	57.3	58.1	58.9
5	39.7	40.1	40.9	41.7	42.5	43.3	44.1	44.9	45.7	46.5	47.3	48.1	48.9	49.7	50.5	51.3	52.1	52.9	53.7	54.5	55.3	56.1	56.9	57.7	58.5
6	39.4	39.8	40.6	41.4	42.2	43.0	43.8	44.6	45.4	46.2	47.0	47.8	48.6	49.4	50.2	51.0	51.8	52.6	53.4	54.2	55.0	55.8	56.6	57.4	58.2
7	39.1	39.5	40.3	41.1	41.9	42.7	43.5	44.3	45.1	45.9	46.7	47.5	48.3	49.1	49.9	50.7	51.5	52.3	53.1	53.9	54.7	55.5	56.3	57.1	57.9
8	38.7	39.1	39.9	40.7	41.5	42.3	43.1	43.9	44.7	45.5	46.3	47.1	47.9	48.7	49.5	50.3	51.1	51.9	52.7	53.5	54.3	55.1	55.9	56.7	57.5
9	38.4	38.8	39.6	40.4	41.2	42.0	42.8	43.6	44.4	45.2	46.0	46.8	47.6	48.4	49.2	50.0	50.8	51.6	52.4	53.2	54.0	54.8	55.6	56.4	57.2
Poi. =	25.0	25.2	25.4	25.6	25.8	26.0	26.2	26.4	26.6	26.8	27.0	27.2	27.4	27.6	27.8	28.0	28.2	28.4	28.6	28.8	29.0	29.2	29.4	29.6	29.8
12.0	51.8	52.2	52.6	53.0	53.4	53.8	54.2	54.6	55.0	55.4	55.8	56.2	56.6	57.0	57.4	57.8	58.2	58.6	59.0	59.4	59.8	60.2	60.6	61.0	61.4
1	51.4	51.8	52.2	52.6	53.0	53.4	53.8	54.2	54.6	55.0	55.4	55.8	56.2	56.6	57.0	57.4	57.8	58.2	58.6	59.0	59.4	59.8	60.2	60.6	61.0
2	50.9	51.3	51.7	52.1	52.5	52.9	53.3	53.7	54.1	54.5	54.9	55.3	55.7	56.1	56.5	56.9	57.3	57.7	58.1	58.5	58.9	59.3	59.7	60.1	60.5
3	50.5	50.9	51.3	51.7	52.1	52.5	52.9	53.3	53.7	54.1	54.5	54.9	55.3	55.7	56.1	56.5	56.9	57.3	57.7	58.1	58.5	58.9	59.3	59.7	60.1
4	50.1	50.5	50.9	51.3	51.7	52.1	52.5	52.9	53.3	53.7	54.1	54.5	54.9	55.3	55.7	56.1	56.5	56.9	57.3	57.7	58.1	58.5	58.9	59.3	59.7
5	49.6	50.0	50.4	50.8	51.2	51.6	52.0	52.4	52.8	53.2	53.6	54.0	54.4	54.8	55.2	55.6	56.0	56.4	56.8	57.2	57.6	58.0	58.4	58.8	59.2
6	49.2	49.6	50.0	50.4	50.8	51.2	51.6	52.0	52.4	52.8	53.2	53.6	54.0	54.4	54.8	55.2	55.6	56.0	56.4	56.8	57.2	57.6	58.0	58.4	58.8
7	48.8	49.2	49.6	50.0	50.4	50.8	51.2	51.6	52.0	52.4	52.8	53.2	53.6	54.0	54.4	54.8	55.2	55.6	56.0	56.4	56.8	57.2	57.6	58.0	58.4
8	48.4	48.8	49.2	49.6	50.0	50.4	50.8	51.2	51.6	52.0	52.4	52.8	53.2	53.6	54.0	54.4	54.8	55.2	55.6	56.0	56.4	56.8	57.2	57.6	58.0
9	48.0	48.4	48.8	49.2	49.6	50.0	50.4	50.8	51.2	51.6	52.0	52.4	52.8	53.2	53.6	54.0	54.4	54.8	55.2	55.6	56.0	56.4	56.8	57.2	57.6

TABLE 39—Continued

TABLE 39—Continued  
EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	15.0	15.2	15.4	15.6	15.8	16.0	16.2	16.4	16.6	16.8	17.0	17.2	17.4	17.6	17.8	18.0	18.2	18.4	18.6	18.8	19.0	19.2	19.4	19.6	19.8
18.0	28.6	29.0	29.3	29.7	30.1	30.5	30.9	31.3	31.6	32.0	32.4	32.8	33.2	33.5	33.9	34.3	34.7	35.1	35.4	35.8	36.2	36.6	37.0	37.3	37.7
.1	28.4	28.8	29.1	29.5	29.9	30.3	30.7	31.1	31.4	31.8	32.2	32.6	33.0	33.3	33.7	34.1	34.5	34.9	35.3	35.7	36.1	36.5	36.9	37.3	37.7
.2	28.1	28.5	28.9	29.3	29.7	30.1	30.5	30.9	31.3	31.6	32.0	32.4	32.8	33.1	33.5	33.9	34.3	34.7	35.1	35.5	35.9	36.3	36.7	37.1	37.5
.3	27.9	28.3	28.6	29.0	29.4	29.8	30.2	30.6	31.0	31.4	31.8	32.2	32.6	33.0	33.4	33.8	34.2	34.6	35.0	35.4	35.8	36.2	36.6	37.0	37.4
.4	27.7	28.1	28.4	28.8	29.2	29.6	30.0	30.4	30.8	31.2	31.6	32.0	32.4	32.8	33.2	33.6	34.0	34.4	34.8	35.2	35.6	36.0	36.4	36.8	37.2
.5	27.5	27.9	28.2	28.6	29.0	29.4	29.8	30.2	30.6	31.0	31.4	31.8	32.2	32.6	33.0	33.4	33.8	34.2	34.6	35.0	35.4	35.8	36.2	36.6	37.0
.6	27.3	27.7	28.0	28.4	28.8	29.2	29.6	30.0	30.4	30.8	31.2	31.6	32.0	32.4	32.8	33.2	33.6	34.0	34.4	34.8	35.2	35.6	36.0	36.4	36.8
.7	27.0	27.4	27.8	28.2	28.6	29.0	29.4	29.8	30.2	30.6	31.0	31.4	31.8	32.2	32.6	33.0	33.4	33.8	34.2	34.6	35.0	35.4	35.8	36.2	36.6
.8	26.8	27.2	27.6	28.0	28.4	28.8	29.2	29.6	30.0	30.4	30.8	31.2	31.6	32.0	32.4	32.8	33.2	33.6	34.0	34.4	34.8	35.2	35.6	36.0	36.4
.9	26.6	27.0	27.4	27.8	28.2	28.6	29.0	29.4	29.8	30.2	30.6	31.0	31.4	31.8	32.2	32.6	33.0	33.4	33.8	34.2	34.6	35.0	35.4	35.8	36.2
Pol. =	20.0	20.2	20.4	20.6	20.8	21.0	21.2	21.4	21.6	21.8	22.0	22.2	22.4	22.6	22.8	23.0	23.2	23.4	23.6	23.8	24.0	24.2	24.4	24.6	24.8
18.0	38.1	38.5	38.9	39.3	39.7	40.1	40.5	40.9	41.3	41.7	42.1	42.5	42.9	43.3	43.7	44.1	44.5	44.9	45.3	45.7	46.1	46.5	46.9	47.3	47.7
.1	37.8	38.2	38.6	39.0	39.4	39.8	40.2	40.6	41.0	41.4	41.8	42.2	42.6	43.0	43.4	43.8	44.2	44.6	45.0	45.4	45.8	46.2	46.6	47.0	47.4
.2	37.5	37.9	38.3	38.7	39.1	39.5	39.9	40.3	40.7	41.1	41.5	41.9	42.3	42.7	43.1	43.5	43.9	44.3	44.7	45.1	45.5	45.9	46.3	46.7	47.1
.3	37.2	37.6	38.0	38.4	38.8	39.2	39.6	40.0	40.4	40.8	41.2	41.6	42.0	42.4	42.8	43.2	43.6	44.0	44.4	44.8	45.2	45.6	46.0	46.4	46.8
.4	36.9	37.3	37.7	38.1	38.5	38.9	39.3	39.7	40.1	40.5	40.9	41.3	41.7	42.1	42.5	42.9	43.3	43.7	44.1	44.5	44.9	45.3	45.7	46.1	46.5
.5	36.6	37.0	37.4	37.8	38.2	38.6	39.0	39.4	39.8	40.2	40.6	41.0	41.4	41.8	42.2	42.6	43.0	43.4	43.8	44.2	44.6	45.0	45.4	45.8	46.2
.6	36.3	36.7	37.1	37.5	37.9	38.3	38.7	39.1	39.5	39.9	40.3	40.7	41.1	41.5	41.9	42.3	42.7	43.1	43.5	43.9	44.3	44.7	45.1	45.5	45.9
.7	36.1	36.5	36.9	37.3	37.7	38.1	38.5	38.9	39.3	39.7	40.1	40.5	40.9	41.3	41.7	42.1	42.5	42.9	43.3	43.7	44.1	44.5	44.9	45.3	45.7
.8	35.8	36.2	36.6	37.0	37.4	37.8	38.2	38.6	39.0	39.4	39.8	40.2	40.6	41.0	41.4	41.8	42.2	42.6	43.0	43.4	43.8	44.2	44.6	45.0	45.4
.9	35.5	35.9	36.3	36.7	37.1	37.5	37.9	38.3	38.7	39.1	39.5	39.9	40.3	40.7	41.1	41.5	41.9	42.3	42.7	43.1	43.5	43.9	44.3	44.7	45.1
Pol. =	25.0	25.2	25.4	25.6	25.8	26.0	26.2	26.4	26.6	26.8	27.0	27.2	27.4	27.6	27.8	28.0	28.2	28.4	28.6	28.8	29.0	29.2	29.4	29.6	29.8
18.0	47.6	48.0	48.4	48.8	49.2	49.6	50.0	50.4	50.8	51.2	51.6	52.0	52.4	52.8	53.2	53.6	54.0	54.4	54.8	55.2	55.6	56.0	56.4	56.8	57.2
.1	47.3	47.7	48.1	48.5	48.9	49.3	49.7	50.1	50.5	50.9	51.3	51.7	52.1	52.5	52.9	53.3	53.7	54.1	54.5	54.9	55.3	55.7	56.1	56.5	56.9
.2	46.9	47.3	47.7	48.1	48.5	48.9	49.3	49.7	50.1	50.5	50.9	51.3	51.7	52.1	52.5	52.9	53.3	53.7	54.1	54.5	54.9	55.3	55.7	56.1	56.5
.3	46.5	46.9	47.3	47.7	48.1	48.5	48.9	49.3	49.7	50.1	50.5	50.9	51.3	51.7	52.1	52.5	52.9	53.3	53.7	54.1	54.5	54.9	55.3	55.7	56.1
.4	46.1	46.5	46.9	47.3	47.7	48.1	48.5	48.9	49.3	49.7	50.1	50.5	50.9	51.3	51.7	52.1	52.5	52.9	53.3	53.7	54.1	54.5	54.9	55.3	55.7
.5	45.8	46.2	46.6	47.0	47.4	47.8	48.2	48.6	49.0	49.4	49.8	50.2	50.6	51.0	51.4	51.8	52.2	52.6	53.0	53.4	53.8	54.2	54.6	55.0	55.4
.6	45.4	45.8	46.2	46.6	47.0	47.4	47.8	48.2	48.6	49.0	49.4	49.8	50.2	50.6	51.0	51.4	51.8	52.2	52.6	53.0	53.4	53.8	54.2	54.6	55.0
.7	45.1	45.5	45.9	46.3	46.7	47.1	47.5	47.9	48.3	48.7	49.1	49.5	49.9	50.3	50.7	51.1	51.5	51.9	52.3	52.7	53.1	53.5	53.9	54.3	54.7
.8	44.7	45.1	45.5	45.9	46.3	46.7	47.1	47.5	47.9	48.3	48.7	49.1	49.5	49.9	50.3	50.7	51.1	51.5	51.9	52.3	52.7	53.1	53.5	53.9	54.3
.9	44.4	44.8	45.2	45.6	46.0	46.4	46.8	47.2	47.6	48.0	48.4	48.8	49.2	49.6	50.0	50.4	50.8	51.2	51.6	52.0	52.4	52.8	53.2	53.6	54.0

TABLE 39—Continued  
EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Poi. =	30.0	30.2	30.4	30.6	30.8	31.0	31.2	31.4	31.6	31.8	32.0	32.2	32.4	32.6	32.8	33.0	33.2	33.4	33.6	33.8	34.0	34.2	34.4	34.6	34.8
13.0	57.2	57.5	57.9	58.3	58.7	59.1	59.5	59.9	60.3	60.7	61.1	61.5	61.9	62.3	62.7	63.1	63.5	63.9	64.3	64.7	65.1	65.5	65.9	66.3	66.7
1	56.7	57.0	57.3	57.6	58.0	58.3	58.7	59.0	59.4	59.8	60.2	60.6	61.0	61.4	61.8	62.2	62.6	63.0	63.4	63.8	64.2	64.6	65.0	65.4	65.8
2	56.3	56.6	56.9	57.2	57.5	57.8	58.1	58.4	58.7	59.0	59.3	59.6	60.0	60.3	60.6	61.0	61.3	61.6	62.0	62.3	62.6	63.0	63.3	63.7	64.0
3	55.8	56.1	56.4	56.7	57.0	57.3	57.6	57.9	58.2	58.5	58.8	59.1	59.4	59.7	60.0	60.3	60.6	61.0	61.3	61.6	62.0	62.3	62.6	63.0	63.3
4	55.3	55.6	55.9	56.2	56.5	56.8	57.1	57.4	57.7	58.0	58.3	58.6	58.9	59.2	59.5	59.8	60.1	60.4	60.7	61.0	61.3	61.6	62.0	62.3	62.6
5	54.8	55.1	55.4	55.7	56.0	56.3	56.6	56.9	57.2	57.5	57.8	58.1	58.4	58.7	59.0	59.3	59.6	60.0	60.3	60.6	60.9	61.2	61.5	61.8	62.1
6	54.3	54.6	54.9	55.2	55.5	55.8	56.1	56.4	56.7	57.0	57.3	57.6	57.9	58.2	58.5	58.8	59.1	59.4	59.7	60.0	60.3	60.6	61.0	61.3	61.6
7	53.8	54.1	54.4	54.7	55.0	55.3	55.6	55.9	56.2	56.5	56.8	57.1	57.4	57.7	58.0	58.3	58.6	59.0	59.3	59.6	59.9	60.2	60.5	60.8	61.1
8	53.3	53.6	53.9	54.2	54.5	54.8	55.1	55.4	55.7	56.0	56.3	56.6	56.9	57.2	57.5	57.8	58.1	58.4	58.7	59.0	59.3	59.6	60.0	60.3	60.6
9	52.8	53.1	53.4	53.7	54.0	54.3	54.6	54.9	55.2	55.5	55.8	56.1	56.4	56.7	57.0	57.3	57.6	57.9	58.2	58.5	58.8	59.1	59.4	59.7	60.0
Poi. =	35.0	35.2	35.4	35.6	35.8	36.0	36.2	36.4	36.6	36.8	37.0	37.2	37.4	37.6	37.8	38.0	38.2	38.4	38.6	38.8	39.0	39.2	39.4	39.6	39.8
13.0	66.7	67.1	67.5	67.9	68.3	68.7	69.1	69.5	69.9	70.3	70.7	71.1	71.5	71.9	72.3	72.7	73.1	73.5	73.9	74.3	74.7	75.1	75.5	75.9	76.3
1	66.2	66.5	66.8	67.1	67.4	67.7	68.0	68.3	68.6	68.9	69.2	69.5	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.4
2	65.6	65.9	66.2	66.5	66.8	67.1	67.4	67.7	68.0	68.3	68.6	68.9	69.2	69.5	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8
3	65.1	65.4	65.7	66.0	66.3	66.6	66.9	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3
4	64.6	64.9	65.2	65.5	65.8	66.1	66.4	66.7	67.0	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8
5	64.1	64.4	64.7	65.0	65.3	65.6	65.9	66.2	66.5	66.8	67.1	67.4	67.7	68.0	68.3	68.6	68.9	69.2	69.5	69.8	70.1	70.4	70.7	71.0	71.3
6	63.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.9	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8
7	63.1	63.4	63.7	64.0	64.3	64.6	64.9	65.2	65.5	65.8	66.1	66.4	66.7	67.0	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3
8	62.6	62.9	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.9	66.2	66.5	66.8	67.1	67.4	67.7	68.0	68.3	68.6	68.9	69.2	69.5	69.8
9	62.1	62.4	62.7	63.0	63.3	63.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.9	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3
Poi. =	40.0	40.2	40.4	40.6	40.8	41.0	41.2	41.4	41.6	41.8	42.0	42.2	42.4	42.6	42.8	43.0	43.2	43.4	43.6	43.8	44.0	44.2	44.4	44.6	44.8
13.0	76.2	76.6	77.0	77.4	77.8	78.2	78.6	79.0	79.4	79.8	80.2	80.6	81.0	81.4	81.8	82.2	82.6	83.0	83.4	83.8	84.2	84.6	85.0	85.4	85.8
1	75.6	76.0	76.4	76.8	77.2	77.6	78.0	78.4	78.8	79.2	79.6	80.0	80.4	80.8	81.2	81.6	82.0	82.4	82.8	83.2	83.6	84.0	84.4	84.8	85.2
2	75.0	75.4	75.8	76.2	76.6	77.0	77.4	77.8	78.2	78.6	79.0	79.4	79.8	80.2	80.6	81.0	81.4	81.8	82.2	82.6	83.0	83.4	83.8	84.2	84.6
3	74.4	74.8	75.2	75.6	76.0	76.4	76.8	77.2	77.6	78.0	78.4	78.8	79.2	79.6	80.0	80.4	80.8	81.2	81.6	82.0	82.4	82.8	83.2	83.6	84.0
4	73.8	74.2	74.6	75.0	75.4	75.8	76.2	76.6	77.0	77.4	77.8	78.2	78.6	79.0	79.4	79.8	80.2	80.6	81.0	81.4	81.8	82.2	82.6	83.0	83.4
5	73.2	73.6	74.0	74.4	74.8	75.2	75.6	76.0	76.4	76.8	77.2	77.6	78.0	78.4	78.8	79.2	79.6	80.0	80.4	80.8	81.2	81.6	82.0	82.4	82.8
6	72.6	73.0	73.4	73.8	74.2	74.6	75.0	75.4	75.8	76.2	76.6	77.0	77.4	77.8	78.2	78.6	79.0	79.4	79.8	80.2	80.6	81.0	81.4	81.8	82.2
7	72.0	72.4	72.8	73.2	73.6	74.0	74.4	74.8	75.2	75.6	76.0	76.4	76.8	77.2	77.6	78.0	78.4	78.8	79.2	79.6	80.0	80.4	80.8	81.2	81.6
8	71.4	71.8	72.2	72.6	73.0	73.4	73.8	74.2	74.6	75.0	75.4	75.8	76.2	76.6	77.0	77.4	77.8	78.2	78.6	79.0	79.4	79.8	80.2	80.6	81.0
9	70.8	71.2	71.6	72.0	72.4	72.8	73.2	73.6	74.0	74.4	74.8	75.2	75.6	76.0	76.4	76.8	77.2	77.6	78.0	78.4	78.8	79.2	79.6	80.0	80.4

TABLE 39—Continued  
EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	20.0	20.2	20.4	20.6	20.8	21.0	21.2	21.4	21.6	21.8	22.0	22.2	22.4	22.6	22.8	23.0	23.2	23.4	23.6	23.8	24.0	24.2	24.4	24.6	24.8
14.0	35.235	6.39	0.39	3.39	7.37	0.37	4.37	7.38	1.38	4.38	8.39	1.39	5.39	8.40	2.40	5.40	9.41	2.41	6.41	9.42	3.42	6.43	0.43	4.43	7
.1	35.035	8.85	7.36	0.36	4.36	7.37	1.37	4.37	8.38	1.38	5.38	8.39	2.39	5.39	9.40	2.40	6.40	9.41	3.41	6.41	4.42	0.42	3.42	7.43	0.43
.2	34.735	1.35	4.35	8.36	1.36	5.36	8.37	2.37	5.37	8.38	2.38	5.38	8.39	2.39	6.40	3.40	6.41	0.41	3.41	7.42	0.42	4.43	7.43	0.43	1
.3	34.534	8.35	2.35	5.35	8.36	3.36	6.37	0.37	3.37	6.38	3.38	6.38	9.39	3.39	6.40	0.40	3.40	7.41	0.41	4.41	7.42	0.42	4.43	7	
.4	34.334	6.34	9.35	2.35	6.35	9.36	3.36	6.36	7.37	0.37	4.37	8.38	1.38	5.38	8.39	7.40	0.40	4.41	7.42	0.42	4.43	7.43	0.43	1	
.5	34.034	3.84	6.35	0.35	3.35	7.36	0.36	3.36	7.37	0.37	4.37	8.38	1.38	5.38	8.39	7.40	0.40	4.41	7.42	0.42	4.43	7.43	0.43	1	
.6	33.734	1.84	4.34	7.35	1.35	4.35	7.36	1.36	4.36	8.37	1.37	4.37	8.38	1.38	4.38	8.39	1.39	4.39	8.40	1.40	5.40	8.41	1.41	5.41	8
.7	33.533	3.84	1.84	5.34	8.35	1.85	5.35	8.36	3.36	6.37	2.37	5.37	8.38	2.38	5.38	8.39	2.39	5.40	8.41	2.41	5.42	8.43	2.43	5.44	8
.8	33.233	6.33	9.34	2.34	6.34	9.35	2.35	6.35	9.36	3.36	6.37	2.37	5.37	8.38	2.38	5.38	8.39	2.39	5.40	8.41	2.41	5.42	8.43	2.43	5
.9	33.033	3.33	6.34	0.34	3.34	6.35	0.35	3.35	6.36	0.36	3.36	6.37	0.37	3.37	6.38	0.38	3.38	6.39	0.39	3.39	6.40	0.40	3.40	6.41	0
Pol. =	25.0	25.2	25.4	25.6	25.8	26.0	26.2	26.4	26.6	26.8	27.0	27.2	27.4	27.6	27.8	28.0	28.2	28.4	28.6	28.8	29.0	29.2	29.4	29.6	29.8
14.0	44.144	4.44	8.45	1.45	5.45	8.46	2.46	5.46	9.47	2.47	0.47	9.48	3.48	6.49	0.49	3.49	7.50	0.50	4.50	8.51	1.51	5.51	8.52	2.52	5
.1	43.744	1.44	4.44	8.45	1.45	5.45	8.46	2.46	5.46	9.47	2.47	0.47	9.48	3.48	6.49	0.49	3.49	7.50	0.50	4.50	8.51	1.51	5.51	8.52	1
.2	43.443	7.44	1.44	4.44	8.45	1.45	5.45	8.46	2.46	5.46	9.47	2.47	0.47	9.48	3.48	6.49	0.49	3.49	7.50	0.50	4.50	8.51	1.51	5.51	7
.3	43.143	4.43	8.44	1.44	5.44	8.45	2.45	5.45	8.46	2.46	5.46	9.47	2.47	0.47	9.48	3.48	6.49	0.49	3.49	7.50	0.50	4.50	8.51	1.51	4
.4	42.843	1.43	4.43	8.44	1.44	5.44	8.45	2.45	5.45	8.46	2.46	5.46	9.47	2.47	0.47	9.48	3.48	6.49	0.49	3.49	7.50	0.50	4.50	8.51	3
.5	42.543	8.43	1.43	5.43	8.44	2.44	5.44	8.45	2.45	5.45	8.46	2.46	5.46	9.47	2.47	0.47	9.48	3.48	6.49	0.49	3.49	7.50	0.50	4.50	2
.6	42.242	5.42	8.43	1.43	5.43	8.44	2.44	5.44	8.45	2.45	5.45	8.46	2.46	5.46	9.47	2.47	0.47	9.48	3.48	6.49	0.49	3.49	7.50	0.50	1
.7	41.942	2.42	5.42	8.43	2.43	5.43	8.44	2.44	5.44	8.45	2.45	5.45	8.46	2.46	5.46	9.47	2.47	0.47	9.48	3.48	6.49	0.49	3.49	7.50	0
.8	41.641	9.42	2.42	5.42	8.43	2.43	5.43	8.44	2.44	5.44	8.45	2.45	5.45	8.46	2.46	5.46	9.47	2.47	0.47	9.48	3.48	6.49	0.49	3.49	2
.9	41.341	6.41	9.42	2.42	5.42	8.43	2.43	5.43	8.44	2.44	5.44	8.45	2.45	5.45	8.46	2.46	5.46	9.47	2.47	0.47	9.48	3.48	6.49	0.49	1
Pol. =	30.0	30.2	30.4	30.6	30.8	31.0	31.2	31.4	31.6	31.8	32.0	32.2	32.4	32.6	32.8	33.0	33.2	33.4	33.6	33.8	34.0	34.2	34.4	34.6	34.8
14.0	52.953	2.53	6.53	9.54	3.54	6.55	0.55	3.55	7.56	0.56	4.56	7.57	1.57	5.57	8.58	2.58	5.58	9.59	2.59	6.59	9.60	3.60	6.61	0.61	3
.1	52.752	8.53	2.53	6.54	9.54	3.55	0.55	3.55	7.56	0.56	4.56	7.57	1.57	5.57	8.58	2.58	5.58	9.59	2.59	6.59	9.60	3.60	6.61	0.61	2
.2	52.552	4.52	8.53	1.53	5.53	8.54	2.54	5.54	9.55	3.55	6.56	2.56	5.56	9.57	3.57	6.58	0.58	3.58	7.59	0.59	4.59	7.60	1.60	4	
.3	51.752	0.52	4.52	7.53	1.53	4.53	8.54	1.54	5.54	8.55	1.55	5.55	8.56	2.56	5.56	9.57	2.57	5.57	8.58	2.58	5.58	9.59	2.59	6	
.4	51.351	7.51	0.52	3.52	7.53	4.53	7.54	1.54	4.54	7.55	1.55	4.55	7.56	2.56	5.56	9.57	2.57	5.57	8.58	2.58	5.58	9.59	2.59	6	
.5	50.951	3.51	6.52	0.52	3.52	7.53	0.53	3.53	7.54	0.54	3.54	7.55	0.55	3.55	7.56	0.56	3.56	7.57	0.57	3.57	7.58	0.58	3.58	7	
.6	50.650	9.51	2.51	6.51	9.52	3.52	6.52	9.53	3.53	6.54	3.54	6.55	9.56	3.55	6.56	9.57	2.57	5.57	8.58	2.58	5.58	9.59	2.59	6	
.7	50.250	5.50	9.51	2.51	6.51	9.52	2.52	6.52	9.53	3.53	6.54	2.54	6.55	9.56	3.55	6.56	9.57	2.57	5.57	8.58	2.58	5.58	9.59	2.59	5
.8	49.950	2.50	5.50	8.51	2.51	6.51	9.52	2.52	6.52	9.53	3.53	6.54	2.54	6.55	9.56	3.55	6.56	9.57	2.57	5.57	8.58	2.58	5.58	9.59	4
.9	49.549	8.50	2.50	5.50	8.51	2.51	6.51	9.52	2.52	6.52	9.53	3.53	6.54	2.54	6.55	9.56	3.55	6.56	9.57	2.57	5.57	8.58	2.58	5.58	3



TABLE 39—Continued  
EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	35.0	35.2	35.4	35.6	35.8	36.0	36.2	36.4	36.6	36.8	37.0	37.2	37.4	37.6	37.8	38.0	38.2	38.4	38.6	38.8	39.0	39.2	39.4	39.6	39.8	40.0
14.0	61.7	62.0	62.4	62.7	63.1	63.4	63.8	64.1	64.5	64.9	65.2	65.6	65.9	66.3	66.7	67.0	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	70.0
1	61.2	61.5	61.9	62.2	62.6	63.0	63.3	63.6	64.0	64.3	64.7	65.0	65.3	65.7	66.0	66.3	66.6	66.9	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3
2	60.8	61.1	61.5	61.8	62.2	62.5	62.9	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.9	66.2	66.5	66.8	67.1	67.4	67.7	68.0	68.3	68.6
3	60.3	60.6	61.0	61.3	61.7	62.0	62.3	62.6	63.0	63.3	63.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.9	67.2	67.5	67.8	68.1
4	59.8	60.1	60.5	60.8	61.1	61.4	61.7	62.0	62.3	62.6	62.9	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.9	66.2	66.5	66.8	67.1	67.4
5	59.3	59.6	60.0	60.3	60.6	60.9	61.2	61.5	61.8	62.1	62.4	62.7	63.0	63.3	63.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.9
6	58.8	59.1	59.5	59.8	60.1	60.4	60.7	61.0	61.3	61.6	61.9	62.2	62.5	62.8	63.1	63.4	63.7	64.0	64.3	64.6	64.9	65.2	65.5	65.8	66.1	66.4
7	58.3	58.6	59.0	59.3	59.6	59.9	60.2	60.5	60.8	61.1	61.4	61.7	62.0	62.3	62.6	62.9	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.9
8	57.8	58.1	58.4	58.7	59.0	59.3	59.6	59.9	60.2	60.5	60.8	61.1	61.4	61.7	62.0	62.3	62.6	62.9	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3
9	57.3	57.6	57.9	58.2	58.5	58.8	59.1	59.4	59.7	60.0	60.3	60.6	60.9	61.2	61.5	61.8	62.1	62.4	62.7	63.0	63.3	63.6	63.9	64.2	64.5	64.8
Pol. =	40.0	40.2	40.4	40.6	40.8	41.0	41.2	41.4	41.6	41.8	42.0	42.2	42.4	42.6	42.8	43.0	43.2	43.4	43.6	43.8	44.0	44.2	44.4	44.6	44.8	45.0
14.0	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.6	75.9	76.2	76.5	76.8	77.1	77.4	77.7	78.0
1	69.9	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.8	75.1	75.4	75.7	76.0	76.3	76.6	76.9	77.2	77.5
2	69.4	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0	74.3	74.6	74.9	75.2	75.5	75.8	76.1	76.4	76.7	77.0
3	68.9	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.6	75.9	76.2	76.5
4	68.4	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.8	75.1	75.4	75.7	76.0
5	67.9	68.3	68.6	68.9	69.2	69.5	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0	74.3	74.6	74.9	75.2	75.5
6	67.4	67.8	68.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.1	74.4	74.7	75.0
7	66.9	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5
8	66.4	66.8	67.1	67.4	67.7	68.0	68.3	68.6	68.9	69.2	69.5	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0
9	65.9	66.3	66.6	66.9	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2	73.5
Pol. =	45.0	45.2	45.4	45.6	45.8	46.0	46.2	46.4	46.6	46.8	47.0	47.2	47.4	47.6	47.8	48.0	48.2	48.4	48.6	48.8	49.0	49.2	49.4	49.6	49.8	50.0
14.0	79.3	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1	85.4	85.7	86.0	86.3	86.6	86.9
1	78.7	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1	85.4	85.7	86.0	86.3
2	78.1	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1	85.4	85.7
3	77.5	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1
4	76.9	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	84.2	84.5
5	76.3	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9
6	75.7	76.1	76.4	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3
7	75.1	75.5	75.8	76.1	76.4	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7
8	74.5	74.9	75.2	75.5	75.8	76.1	76.4	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1
9	74.0	74.3	74.6	74.9	75.2	75.5	75.8	76.1	76.4	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5

TABLE 39—Continued  
EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	30.0	30.2	30.4	30.6	30.8	31.0	31.2	31.4	31.6	31.8	32.0	32.2	32.4	32.6	32.8	33.0	33.2	33.4	33.6	33.8	34.0	34.2	34.4	34.6	34.8
15.0	49.1	49.5	49.8	50.1	50.5	50.8	51.1	51.4	51.8	52.2	52.5	52.8	53.1	53.4	53.7	54.1	54.4	54.7	55.0	55.4	55.7	56.0	56.4	56.7	57.0
.1	48.8	49.1	49.4	49.7	50.0	50.3	50.6	50.9	51.2	51.5	51.8	52.1	52.4	52.7	53.0	53.3	53.6	53.9	54.2	54.5	54.8	55.1	55.4	55.7	56.0
.2	48.5	48.8	49.1	49.4	49.7	50.0	50.3	50.6	50.9	51.2	51.5	51.8	52.1	52.4	52.7	53.0	53.3	53.6	53.9	54.2	54.5	54.8	55.1	55.4	55.7
.3	48.1	48.4	48.7	49.0	49.3	49.6	49.9	50.2	50.5	50.8	51.1	51.4	51.7	52.0	52.3	52.6	52.9	53.2	53.5	53.8	54.1	54.4	54.7	55.0	55.3
.4	47.8	48.1	48.4	48.7	49.0	49.3	49.6	49.9	50.2	50.5	50.8	51.1	51.4	51.7	52.0	52.3	52.6	52.9	53.2	53.5	53.8	54.1	54.4	54.7	55.0
.5	47.5	47.8	48.1	48.4	48.7	49.0	49.3	49.6	49.9	50.2	50.5	50.8	51.1	51.4	51.7	52.0	52.3	52.6	52.9	53.2	53.5	53.8	54.1	54.4	54.7
.6	47.1	47.4	47.7	48.0	48.3	48.6	48.9	49.2	49.5	49.8	50.1	50.4	50.7	51.0	51.3	51.6	51.9	52.2	52.5	52.8	53.1	53.4	53.7	54.0	54.3
.7	46.8	47.1	47.4	47.7	48.0	48.3	48.6	48.9	49.2	49.5	49.8	50.1	50.4	50.7	51.0	51.3	51.6	51.9	52.2	52.5	52.8	53.1	53.4	53.7	54.0
.8	46.5	46.8	47.1	47.4	47.7	48.0	48.3	48.6	48.9	49.2	49.5	49.8	50.1	50.4	50.7	51.0	51.3	51.6	51.9	52.2	52.5	52.8	53.1	53.4	53.7
.9	46.2	46.5	46.8	47.1	47.4	47.7	48.0	48.3	48.6	48.9	49.2	49.5	49.8	50.1	50.4	50.7	51.0	51.3	51.6	51.9	52.2	52.5	52.8	53.1	53.4
Pol. =	35.0	35.2	35.4	35.6	35.8	36.0	36.2	36.4	36.6	36.8	37.0	37.2	37.4	37.6	37.8	38.0	38.2	38.4	38.6	38.8	39.0	39.2	39.4	39.6	39.8
15.0	57.3	57.7	58.0	58.3	58.6	58.9	59.2	59.5	59.8	60.1	60.4	60.7	61.0	61.3	61.6	61.9	62.2	62.5	62.8	63.1	63.4	63.7	64.0	64.3	64.6
.1	56.9	57.3	57.6	57.9	58.2	58.5	58.8	59.1	59.4	59.7	60.0	60.3	60.6	60.9	61.2	61.5	61.8	62.1	62.4	62.7	63.0	63.3	63.6	63.9	64.2
.2	56.5	56.9	57.2	57.5	57.8	58.1	58.4	58.7	59.0	59.3	59.6	59.9	60.2	60.5	60.8	61.1	61.4	61.7	62.0	62.3	62.6	62.9	63.2	63.5	63.8
.3	56.1	56.5	56.8	57.1	57.4	57.7	58.0	58.3	58.6	58.9	59.2	59.5	59.8	60.1	60.4	60.7	61.0	61.3	61.6	61.9	62.2	62.5	62.8	63.1	63.4
.4	55.8	56.1	56.4	56.7	57.0	57.3	57.6	57.9	58.2	58.5	58.8	59.1	59.4	59.7	60.0	60.3	60.6	60.9	61.2	61.5	61.8	62.1	62.4	62.7	63.0
.5	55.4	55.7	56.0	56.3	56.6	56.9	57.2	57.5	57.8	58.1	58.4	58.7	59.0	59.3	59.6	59.9	60.2	60.5	60.8	61.1	61.4	61.7	62.0	62.3	62.6
.6	55.0	55.3	55.6	55.9	56.2	56.5	56.8	57.1	57.4	57.7	58.0	58.3	58.6	58.9	59.2	59.5	59.8	60.1	60.4	60.7	61.0	61.3	61.6	61.9	62.2
.7	54.6	54.9	55.2	55.5	55.8	56.1	56.4	56.7	57.0	57.3	57.6	57.9	58.2	58.5	58.8	59.1	59.4	59.7	60.0	60.3	60.6	60.9	61.2	61.5	61.8
.8	54.3	54.6	54.9	55.2	55.5	55.8	56.1	56.4	56.7	57.0	57.3	57.6	57.9	58.2	58.5	58.8	59.1	59.4	59.7	60.0	60.3	60.6	60.9	61.2	61.5
.9	53.9	54.2	54.5	54.8	55.1	55.4	55.7	56.0	56.3	56.6	56.9	57.2	57.5	57.8	58.1	58.4	58.7	59.0	59.3	59.6	59.9	60.2	60.5	60.8	61.1
Pol. =	40.0	40.2	40.4	40.6	40.8	41.0	41.2	41.4	41.6	41.8	42.0	42.2	42.4	42.6	42.8	43.0	43.2	43.4	43.6	43.8	44.0	44.2	44.4	44.6	44.8
15.0	65.5	65.9	66.2	66.5	66.8	67.1	67.4	67.7	68.0	68.3	68.6	68.9	69.2	69.5	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8
.1	65.1	65.5	65.8	66.1	66.4	66.7	67.0	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4
.2	64.8	65.2	65.5	65.8	66.1	66.4	66.7	67.0	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1
.3	64.4	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.9	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7
.4	63.9	64.3	64.6	64.9	65.2	65.5	65.8	66.1	66.4	66.7	67.0	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2
.5	63.5	63.9	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.9	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8
.6	62.9	63.3	63.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.9	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2
.7	62.4	62.7	63.0	63.3	63.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.9	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.6
.8	62.0	62.3	62.6	62.9	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.9	66.2	66.5	66.8	67.1	67.4	67.7	68.0	68.3	68.6	68.9	69.2
.9	61.6	61.9	62.2	62.5	62.8	63.1	63.4	63.7	64.0	64.3	64.6	64.9	65.2	65.5	65.8	66.1	66.4	66.7	67.0	67.3	67.6	67.9	68.2	68.5	68.8

TABLE 39—Continued  
EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol =	45.0	45.2	45.4	45.6	45.8	46.0	46.2	46.4	46.6	46.8	47.0	47.2	47.4	47.6	47.8	48.0	48.2	48.4	48.6	48.8	49.0	49.2	49.4	49.6	49.8
15.0	73.7	74.0	74.4	74.7	75.0	75.4	75.7	76.0	76.3	76.7	77.0	77.3	77.7	78.0	78.3	78.6	79.0	79.3	79.6	79.9	80.3	80.6	80.9	81.3	81.6
1	73.2	73.5	73.9	74.2	74.5	74.8	75.1	75.4	75.7	76.0	76.3	76.7	77.0	77.3	77.7	78.0	78.3	78.6	78.9	79.2	79.5	79.8	80.1	80.4	80.7
2	72.7	73.0	73.3	73.7	74.0	74.3	74.6	75.0	75.3	75.6	75.9	76.2	76.6	76.9	77.2	77.5	77.8	78.1	78.4	78.7	79.0	79.3	79.6	80.0	80.3
3	72.2	72.5	72.8	73.1	73.4	73.7	74.0	74.4	74.7	75.0	75.3	75.6	76.0	76.3	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7
4	71.7	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.6	76.0	76.3	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1
5	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.8	75.1	75.4	75.7	76.0	76.3	76.7	77.0	77.3	77.6	77.9	78.2	78.5
6	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0	74.3	74.6	74.9	75.2	75.5	75.8	76.1	76.4	76.7	77.0	77.3	77.6	77.9
7	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.6	75.9	76.2	76.5	76.8	77.1	77.4
8	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.8	75.1	75.4	75.7	76.0	76.3	76.6	76.9
9	69.2	69.5	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0	74.3	74.6	74.9	75.2	75.5	75.8	76.1	76.4
Pol =	50.0	50.2	50.4	50.6	50.8	51.0	51.2	51.4	51.6	51.8	52.0	52.2	52.4	52.6	52.8	53.0	53.2	53.4	53.6	53.8	54.0	54.2	54.4	54.6	54.8
15.0	81.3	82.2	83.2	84.2	85.2	86.2	87.2	88.2	89.2	90.2	91.2	92.2	93.2	94.2	95.2	96.2	97.2	98.2	99.2	100.2	101.2	102.2	103.2	104.2	105.2
1	81.3	81.7	82.1	82.5	82.9	83.3	83.7	84.1	84.5	84.9	85.3	85.7	86.1	86.5	86.9	87.3	87.7	88.1	88.5	88.9	89.3	89.7	90.1	90.5	90.9
2	80.8	81.1	81.4	81.7	82.0	82.3	82.6	82.9	83.2	83.5	83.8	84.1	84.4	84.7	85.0	85.3	85.6	85.9	86.2	86.5	86.8	87.1	87.4	87.7	88.0
3	80.2	80.5	80.8	81.1	81.4	81.7	82.0	82.3	82.6	82.9	83.2	83.5	83.8	84.1	84.4	84.7	85.0	85.3	85.6	85.9	86.2	86.5	86.8	87.1	87.4
4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1	85.4	85.7	86.0	86.3	86.6	86.9
5	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1	85.4	85.7	86.0	86.3
6	78.6	78.9	79.2	79.5	79.8	80.1	80.4	80.7	81.0	81.3	81.6	81.9	82.2	82.5	82.8	83.1	83.4	83.7	84.0	84.3	84.6	84.9	85.2	85.5	85.8
7	78.0	78.3	78.6	78.9	79.2	79.5	79.8	80.1	80.4	80.7	81.0	81.3	81.6	81.9	82.2	82.5	82.8	83.1	83.4	83.7	84.0	84.3	84.6	84.9	85.2
8	77.5	77.8	78.1	78.4	78.7	79.0	79.3	79.6	79.9	80.2	80.5	80.8	81.1	81.4	81.7	82.0	82.3	82.6	82.9	83.2	83.5	83.8	84.1	84.4	84.7
9	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	84.2
Pol =	55.0	55.2	55.4	55.6	55.8	56.0	56.2	56.4	56.6	56.8	57.0	57.2	57.4	57.6	57.8	58.0	58.2	58.4	58.6	58.8	59.0	59.2	59.4	59.6	59.8
15.0	90.1	90.4	90.7	91.0	91.3	91.6	91.9	92.2	92.5	92.8	93.1	93.4	93.7	94.0	94.3	94.6	94.9	95.2	95.5	95.8	96.1	96.4	96.7	97.0	97.3
1	89.5	89.8	90.1	90.4	90.7	91.0	91.3	91.6	91.9	92.2	92.5	92.8	93.1	93.4	93.7	94.0	94.3	94.6	94.9	95.2	95.5	95.8	96.1	96.4	96.7
2	88.9	89.2	89.5	89.8	90.1	90.4	90.7	91.0	91.3	91.6	91.9	92.2	92.5	92.8	93.1	93.4	93.7	94.0	94.3	94.6	94.9	95.2	95.5	95.8	96.1
3	88.2	88.5	88.8	89.1	89.4	89.7	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.7	93.0	93.3	93.6	93.9	94.2	94.5	94.8	95.1	95.4
4	87.6	87.9	88.2	88.5	88.8	89.1	89.4	89.7	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.7	93.0	93.3	93.6	93.9	94.2	94.5	94.8
5	87.0	87.3	87.6	87.9	88.2	88.5	88.8	89.1	89.4	89.7	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.7	93.0	93.3	93.6	93.9	94.2
6	86.4	86.7	87.0	87.3	87.6	87.9	88.2	88.5	88.8	89.1	89.4	89.7	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.7	93.0	93.3	93.6
7	85.8	86.1	86.4	86.7	87.0	87.3	87.6	87.9	88.2	88.5	88.8	89.1	89.4	89.7	90.0	90.3	90.6	90.9	91.2	91.5	91.8	92.1	92.4	92.7	93.0
8	85.3	85.6	85.9	86.2	86.5	86.8	87.1	87.4	87.7	88.0	88.3	88.6	88.9	89.2	89.5	89.8	90.1	90.4	90.7	91.0	91.3	91.6	91.9	92.2	92.5
9	84.7	85.0	85.3	85.6	85.9	86.2	86.5	86.8	87.1	87.4	87.7	88.0	88.3	88.6	88.9	89.2	89.5	89.8	90.1	90.4	90.7	91.0	91.3	91.6	91.9

TABLE 39—Continued  
EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Poi. =	35.0	36.2	35.4	35.6	35.8	36.0	36.2	36.4	36.6	36.8	37.0	37.2	37.4	37.6	37.8	38.0	38.2	38.4	38.6	38.8	39.0	39.2	39.4	39.6	39.8
16.0	53.5	53.8	54.1	54.4	54.7	55.0	55.3	55.6	55.9	56.2	56.5	56.8	57.1	57.4	57.7	58.0	58.3	58.6	58.9	59.2	59.5	59.8	60.1	60.4	60.7
1.1	52.2	52.5	52.8	53.1	53.4	53.7	54.0	54.3	54.6	54.9	55.2	55.5	55.8	56.1	56.4	56.7	57.0	57.3	57.6	57.9	58.2	58.5	58.8	59.1	59.4
2.2	51.8	52.1	52.4	52.7	53.0	53.3	53.6	53.9	54.2	54.5	54.8	55.1	55.4	55.7	56.0	56.3	56.6	56.9	57.2	57.5	57.8	58.1	58.4	58.7	59.0
3.3	51.4	51.7	52.0	52.3	52.6	52.9	53.2	53.5	53.8	54.1	54.4	54.7	55.0	55.3	55.6	55.9	56.2	56.5	56.8	57.1	57.4	57.7	58.0	58.3	58.6
4.4	51.0	51.3	51.6	51.9	52.2	52.5	52.8	53.1	53.4	53.7	54.0	54.3	54.6	54.9	55.2	55.5	55.8	56.1	56.4	56.7	57.0	57.3	57.6	57.9	58.2
5.5	50.6	50.9	51.2	51.5	51.8	52.1	52.4	52.7	53.0	53.3	53.6	53.9	54.2	54.5	54.8	55.1	55.4	55.7	56.0	56.3	56.6	56.9	57.2	57.5	57.8
6.6	50.2	50.5	50.8	51.1	51.4	51.7	52.0	52.3	52.6	52.9	53.2	53.5	53.8	54.1	54.4	54.7	55.0	55.3	55.6	55.9	56.2	56.5	56.8	57.1	57.4
7.7	49.8	50.1	50.4	50.7	51.0	51.3	51.6	51.9	52.2	52.5	52.8	53.1	53.4	53.7	54.0	54.3	54.6	54.9	55.2	55.5	55.8	56.1	56.4	56.7	57.0
8.8	49.4	49.7	50.0	50.3	50.6	50.9	51.2	51.5	51.8	52.1	52.4	52.7	53.0	53.3	53.6	53.9	54.2	54.5	54.8	55.1	55.4	55.7	56.0	56.3	56.6
9.9	49.0	49.3	49.6	49.9	50.2	50.5	50.8	51.1	51.4	51.7	52.0	52.3	52.6	52.9	53.2	53.5	53.8	54.1	54.4	54.7	55.0	55.3	55.6	55.9	56.2
Poi. =	40.0	40.2	40.4	40.6	40.8	41.0	41.2	41.4	41.6	41.8	42.0	42.2	42.4	42.6	42.8	43.0	43.2	43.4	43.6	43.8	44.0	44.2	44.4	44.6	44.8
16.0	61.2	61.5	61.8	62.1	62.4	62.7	63.0	63.3	63.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.9	67.2	67.5	67.8	68.1	68.4
1.1	60.8	61.1	61.4	61.7	62.0	62.3	62.6	62.9	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.9	66.2	66.5	66.8	67.1	67.4	67.7	68.0
2.2	60.4	60.7	61.0	61.3	61.6	61.9	62.2	62.5	62.8	63.1	63.4	63.7	64.0	64.3	64.6	64.9	65.2	65.5	65.8	66.1	66.4	66.7	67.0	67.3	67.6
3.3	60.0	60.3	60.6	60.9	61.2	61.5	61.8	62.1	62.4	62.7	63.0	63.3	63.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	66.0	66.3	66.6	66.9	67.2
4.4	59.6	59.9	60.2	60.5	60.8	61.1	61.4	61.7	62.0	62.3	62.6	62.9	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6	65.9	66.2	66.5	66.8
5.5	59.2	59.5	59.8	60.1	60.4	60.7	61.0	61.3	61.6	61.9	62.2	62.5	62.8	63.1	63.4	63.7	64.0	64.3	64.6	64.9	65.2	65.5	65.8	66.1	66.4
6.6	58.8	59.1	59.4	59.7	60.0	60.3	60.6	60.9	61.2	61.5	61.8	62.1	62.4	62.7	63.0	63.3	63.6	63.9	64.2	64.5	64.8	65.1	65.4	65.7	66.0
7.7	58.4	58.7	59.0	59.3	59.6	59.9	60.2	60.5	60.8	61.1	61.4	61.7	62.0	62.3	62.6	62.9	63.2	63.5	63.8	64.1	64.4	64.7	65.0	65.3	65.6
8.8	58.0	58.3	58.6	58.9	59.2	59.5	59.8	60.1	60.4	60.7	61.0	61.3	61.6	61.9	62.2	62.5	62.8	63.1	63.4	63.7	64.0	64.3	64.6	64.9	65.2
9.9	57.6	57.9	58.2	58.5	58.8	59.1	59.4	59.7	60.0	60.3	60.6	60.9	61.2	61.5	61.8	62.1	62.4	62.7	63.0	63.3	63.6	63.9	64.2	64.5	64.8
Poi. =	45.0	45.2	45.4	45.6	45.8	46.0	46.2	46.4	46.6	46.8	47.0	47.2	47.4	47.6	47.8	48.0	48.2	48.4	48.6	48.8	49.0	49.2	49.4	49.6	49.8
16.0	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.8	75.1	75.4	75.7	76.0
1.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.6
2.2	68.0	68.3	68.6	68.9	69.2	69.5	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0	74.3	74.6	74.9	75.2
3.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.8
4.4	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.1	74.4
5.5	66.8	67.1	67.4	67.7	68.0	68.3	68.6	68.9	69.2	69.5	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0
6.6	66.4	66.7	67.0	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6
7.7	66.0	66.3	66.6	66.9	67.2	67.5	67.8	68.1	68.4	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2
8.8	65.6	65.9	66.2	66.5	66.8	67.1	67.4	67.7	68.0	68.3	68.6	68.9	69.2	69.5	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8
9.9	65.2	65.5	65.8	66.1	66.4	66.7	67.0	67.3	67.6	67.9	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	50.0	50.2	50.4	50.6	50.8	51.0	51.2	51.4	51.6	51.8	52.0	52.2	52.4	52.6	52.8	53.0	53.2	53.4	53.6	53.8	54.0	54.2	54.4	54.6	54.8
16.0	76.576	77.1	77.4	77.7	77.8	78.3	78.6	78.9	79.2	79.5	79.8	80.2	80.5	80.8	81.1	81.4	81.7	82.0	82.3	82.6	82.9	83.2	83.5	83.8	84.1
1	76.076	76.6	76.9	77.2	77.5	77.8	78.1	78.4	78.7	79.0	79.3	79.6	79.9	80.2	80.5	80.8	81.1	81.4	81.7	82.0	82.3	82.6	82.9	83.2	83.5
2	75.576	76.1	76.4	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0
3	75.076	75.6	75.9	76.2	76.5	76.8	77.1	77.4	77.7	78.0	78.3	78.6	78.9	79.2	79.5	79.8	80.1	80.4	80.7	81.0	81.3	81.6	81.9	82.2	82.5
4	74.574	75.1	75.4	75.7	76.0	76.3	76.6	76.9	77.2	77.5	77.8	78.1	78.4	78.7	79.0	79.3	79.6	79.9	80.2	80.5	80.8	81.1	81.4	81.7	82.0
5	74.074	74.6	74.9	75.2	75.5	75.8	76.1	76.4	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5
6	73.573	74.1	74.4	74.7	75.0	75.3	75.6	75.9	76.2	76.5	76.8	77.1	77.4	77.7	78.0	78.3	78.6	78.9	79.2	79.5	79.8	80.1	80.4	80.7	81.0
7	73.073	73.6	73.9	74.2	74.5	74.8	75.1	75.4	75.7	76.0	76.3	76.6	76.9	77.2	77.5	77.8	78.1	78.4	78.7	79.0	79.3	79.6	79.9	80.2	80.5
8	72.572	73.1	73.4	73.7	74.0	74.3	74.6	74.9	75.2	75.5	75.8	76.1	76.4	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0
9	72.072	72.6	72.9	73.2	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.6	75.9	76.2	76.5	76.8	77.1	77.4	77.7	78.0	78.3	78.6	78.9	79.2	79.5
Pol. =	55.0	55.2	55.4	55.6	55.8	56.0	56.2	56.4	56.6	56.8	57.0	57.2	57.4	57.6	57.8	58.0	58.2	58.4	58.6	58.8	59.0	59.2	59.4	59.6	59.8
16.0	84.184	84.7	85.0	85.3	85.6	85.9	86.2	86.5	86.8	87.1	87.4	87.7	88.0	88.3	88.6	88.9	89.2	89.5	89.8	90.1	90.4	90.7	91.0	91.3	91.6
1	83.683	84.2	84.5	84.8	85.1	85.4	85.7	86.0	86.3	86.6	86.9	87.2	87.5	87.8	88.1	88.4	88.7	89.0	89.3	89.6	89.9	90.2	90.5	90.8	91.1
2	83.183	83.7	84.0	84.3	84.6	84.9	85.2	85.5	85.8	86.1	86.4	86.7	87.0	87.3	87.6	87.9	88.2	88.5	88.8	89.1	89.4	89.7	90.0	90.3	90.6
3	82.682	83.2	83.5	83.8	84.1	84.4	84.7	85.0	85.3	85.6	85.9	86.2	86.5	86.8	87.1	87.4	87.7	88.0	88.3	88.6	88.9	89.2	89.5	89.8	90.1
4	82.181	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1	85.4	85.7	86.0	86.3	86.6	86.9	87.2	87.5	87.8	88.1	88.4	88.7	89.0	89.3	89.6
5	81.681	82.2	82.5	82.8	83.1	83.4	83.7	84.0	84.3	84.6	84.9	85.2	85.5	85.8	86.1	86.4	86.7	87.0	87.3	87.6	87.9	88.2	88.5	88.8	89.1
6	81.181	81.7	82.0	82.3	82.6	82.9	83.2	83.5	83.8	84.1	84.4	84.7	85.0	85.3	85.6	85.9	86.2	86.5	86.8	87.1	87.4	87.7	88.0	88.3	88.6
7	80.680	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1	85.4	85.7	86.0	86.3	86.6	86.9	87.2	87.5	87.8	88.1
8	80.180	80.7	81.0	81.3	81.6	81.9	82.2	82.5	82.8	83.1	83.4	83.7	84.0	84.3	84.6	84.9	85.2	85.5	85.8	86.1	86.4	86.7	87.0	87.3	87.6
9	79.679	80.2	80.5	80.8	81.1	81.4	81.7	82.0	82.3	82.6	82.9	83.2	83.5	83.8	84.1	84.4	84.7	85.0	85.3	85.6	85.9	86.2	86.5	86.8	87.1
Pol. =	60.0	60.2	60.4	60.6	60.8	61.0	61.2	61.4	61.6	61.8	62.0	62.2	62.4	62.6	62.8	63.0	63.2	63.4	63.6	63.8	64.0	64.2	64.4	64.6	64.8
16.0	91.892	91.9	92.0	92.1	92.2	92.3	92.4	92.5	92.6	92.7	92.8	92.9	93.0	93.1	93.2	93.3	93.4	93.5	93.6	93.7	93.8	93.9	94.0	94.1	94.2
1	91.391	91.4	91.5	91.6	91.7	91.8	91.9	92.0	92.1	92.2	92.3	92.4	92.5	92.6	92.7	92.8	92.9	93.0	93.1	93.2	93.3	93.4	93.5	93.6	93.7
2	90.890	90.9	91.0	91.1	91.2	91.3	91.4	91.5	91.6	91.7	91.8	91.9	92.0	92.1	92.2	92.3	92.4	92.5	92.6	92.7	92.8	92.9	93.0	93.1	93.2
3	90.390	90.4	90.5	90.6	90.7	90.8	90.9	91.0	91.1	91.2	91.3	91.4	91.5	91.6	91.7	91.8	91.9	92.0	92.1	92.2	92.3	92.4	92.5	92.6	92.7
4	89.889	89.9	90.0	90.1	90.2	90.3	90.4	90.5	90.6	90.7	90.8	90.9	91.0	91.1	91.2	91.3	91.4	91.5	91.6	91.7	91.8	91.9	92.0	92.1	92.2
5	89.389	89.4	89.5	89.6	89.7	89.8	89.9	90.0	90.1	90.2	90.3	90.4	90.5	90.6	90.7	90.8	90.9	91.0	91.1	91.2	91.3	91.4	91.5	91.6	91.7
6	88.888	88.9	89.0	89.1	89.2	89.3	89.4	89.5	89.6	89.7	89.8	89.9	90.0	90.1	90.2	90.3	90.4	90.5	90.6	90.7	90.8	90.9	91.0	91.1	91.2
7	88.388	88.4	88.5	88.6	88.7	88.8	88.9	89.0	89.1	89.2	89.3	89.4	89.5	89.6	89.7	89.8	89.9	90.0	90.1	90.2	90.3	90.4	90.5	90.6	90.7
8	87.887	87.9	88.0	88.1	88.2	88.3	88.4	88.5	88.6	88.7	88.8	88.9	89.0	89.1	89.2	89.3	89.4	89.5	89.6	89.7	89.8	89.9	90.0	90.1	90.2
9	87.387	87.4	87.5	87.6	87.7	87.8	87.9	88.0	88.1	88.2	88.3	88.4	88.5	88.6	88.7	88.8	88.9	89.0	89.1	89.2	89.3	89.4	89.5	89.6	89.7

TABLE 39—Continued  
EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

EXPANDED HORNER'S TABLE OF CORRELATIONS																										
Pol. =	45.0	45.2	45.4	45.6	45.8	46.0	46.2	46.4	46.6	46.8	47.0	47.2	47.4	47.6	47.8	48.0	48.2	48.4	48.6	48.8	49.0	49.2	49.4	49.6	49.8	
17.0	64.5	64.8	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6	67.8	68.0	68.2	68.4	68.6	68.8	69.0	69.2	69.4	69.6
1	64.1	64.4	64.6	64.8	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6	67.8	68.0	68.2	68.4	68.6	68.8	69.0	69.2
2	63.7	64.0	64.2	64.4	64.6	64.8	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6	67.8	68.0	68.2	68.4	68.6	68.8
3	63.3	63.6	63.8	64.0	64.2	64.4	64.6	64.8	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6	67.8	68.0	68.2	68.4
4	62.9	63.2	63.4	63.6	63.8	64.0	64.2	64.4	64.6	64.8	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6	67.8	68.0
5	62.5	62.8	63.0	63.2	63.4	63.6	63.8	64.0	64.2	64.4	64.6	64.8	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6
6	62.1	62.4	62.6	62.8	63.0	63.2	63.4	63.6	63.8	64.0	64.2	64.4	64.6	64.8	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2
7	61.7	62.0	62.2	62.4	62.6	62.8	63.0	63.2	63.4	63.6	63.8	64.0	64.2	64.4	64.6	64.8	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8
8	61.3	61.6	61.8	62.0	62.2	62.4	62.6	62.8	63.0	63.2	63.4	63.6	63.8	64.0	64.2	64.4	64.6	64.8	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4
9	61.1	61.3	61.5	61.7	61.9	62.1	62.3	62.5	62.7	62.9	63.1	63.3	63.5	63.7	63.9	64.1	64.3	64.5	64.7	64.9	65.1	65.3	65.5	65.7	65.9	66.1
Pol. =	50.0	50.2	50.4	50.6	50.8	51.0	51.2	51.4	51.6	51.8	52.0	52.2	52.4	52.6	52.8	53.0	53.2	53.4	53.6	53.8	54.0	54.2	54.4	54.6	54.8	
17.0	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0	74.3	74.6	74.9	75.2	75.5	75.8	76.1	76.4	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	
1	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.8	75.1	75.4	75.7	76.0	76.3	76.6	76.9	77.2	77.5	77.8	78.1	78.4	
2	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.6	75.9	76.2	76.5	76.8	77.1	77.4	77.7	78.0	
3	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0	74.3	74.6	74.9	75.2	75.5	75.8	76.1	76.4	76.7	77.0	77.3	77.6	
4	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.6	75.9	76.2	76.5	76.8	77.1	
5	69.5	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0	74.3	74.6	74.9	75.2	75.5	75.8	76.1	76.4	76.7	
6	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.8	75.1	75.4	75.7	76.0	76.3	
7	68.7	69.0	69.3	69.6	69.9	70.2	70.5	70.8	71.1	71.4	71.7	72.0	72.3	72.6	72.9	73.2	73.5	73.8	74.1	74.4	74.7	75.0	75.3	75.6	75.9	
8	68.3	68.6	68.9	69.2	69.5	69.8	70.1	70.4	70.7	71.0	71.3	71.6	71.9	72.2	72.5	72.8	73.1	73.4	73.7	74.0	74.3	74.6	74.9	75.2	75.5	
9	67.9	68.2	68.5	68.8	69.1	69.4	69.7	70.0	70.3	70.6	70.9	71.2	71.5	71.8	72.1	72.4	72.7	73.0	73.3	73.6	73.9	74.2	74.5	74.8	75.1	
Pol. =	55.0	55.2	55.4	55.6	55.8	56.0	56.2	56.4	56.6	56.8	57.0	57.2	57.4	57.6	57.8	58.0	58.2	58.4	58.6	58.8	59.0	59.2	59.4	59.6	59.8	
17.0	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1	85.4	85.7	86.0	
1	78.4	78.7	79.0	79.3	79.6	79.9	80.2	80.5	80.8	81.1	81.4	81.7	82.0	82.3	82.6	82.9	83.2	83.5	83.8	84.1	84.4	84.7	85.0	85.3	85.6	
2	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1	
3	77.5	77.8	78.1	78.4	78.7	79.0	79.3	79.6	79.9	80.2	80.5	80.8	81.1	81.4	81.7	82.0	82.3	82.6	82.9	83.2	83.5	83.8	84.1	84.4	84.7	
4	77.1	77.4	77.7	78.0	78.3	78.6	78.9	79.2	79.5	79.8	80.1	80.4	80.7	81.0	81.3	81.6	81.9	82.2	82.5	82.8	83.1	83.4	83.7	84.0	84.3	
5	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	83.0	83.3	83.6	83.9	
6	76.3	76.6	76.9	77.2	77.5	77.8	78.1	78.4	78.7	79.0	79.3	79.6	79.9	80.2	80.5	80.8	81.1	81.4	81.7	82.0	82.3	82.6	82.9	83.2	83.5	
7	75.9	76.2	76.5	76.8	77.1	77.4	77.7	78.0	78.3	78.6	78.9	79.2	79.5	79.8	80.1	80.4	80.7	81.0	81.3	81.6	81.9	82.2	82.5	82.8	83.1	
8	75.5	75.8	76.1	76.4	76.7	77.0	77.3	77.6	77.9	78.2	78.5	78.8	79.1	79.4	79.7	80.0	80.3	80.6	80.9	81.2	81.5	81.8	82.1	82.4	82.7	
9	75.1	75.4	75.7	76.0	76.3	76.6	76.9	77.2	77.5	77.8	78.1	78.4	78.7	79.0	79.3	79.6	79.9	80.2	80.5	80.8	81.1	81.4	81.7	82.0	82.3	
	74.7	75.0	75.3	75.6	75.9	76.2	76.5	76.8	77.1	77.4	77.7	78.0	78.3	78.6	78.9	79.2	79.5	79.8	80.1	80.4	80.7	81.0	81.3	81.6	81.9	

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	50.0	60.2	60.4	60.6	60.8	61.0	61.2	61.4	61.6	61.8	62.0	62.2	62.4	62.6	62.8	63.0	63.2	63.4	63.6	63.8	64.0	64.2	64.4	64.6	64.8
17.0	86.0	86.3	86.6	86.8	87.1	87.4	87.7	88.0	88.3	88.6	88.8	89.1	89.4	89.7	90.0	90.3	90.6	90.9	91.1	91.4	91.7	92.0	92.3	92.6	92.9
1	85.4	85.7	86.0	86.3	86.6	86.9	87.2	87.4	87.7	88.0	88.3	88.6	88.9	89.1	89.4	89.7	90.0	90.3	90.6	90.9	91.1	91.4	91.7	92.0	92.3
2	85.0	85.2	85.5	85.8	86.1	86.4	86.7	86.9	87.2	87.5	87.8	88.1	88.4	88.6	88.9	89.2	89.5	89.8	90.1	90.3	90.6	90.9	91.2	91.5	91.8
3	84.4	84.7	85.0	85.3	85.6	85.9	86.2	86.4	86.7	87.0	87.3	87.5	87.8	88.1	88.4	88.7	89.0	89.3	89.5	89.8	90.1	90.3	90.6	90.9	91.2
4	83.9	84.2	84.5	84.8	85.1	85.4	85.7	86.0	86.3	86.6	86.9	87.2	87.5	87.8	88.1	88.4	88.7	89.0	89.3	89.5	89.8	90.1	90.3	90.6	90.9
5	83.4	83.7	84.0	84.3	84.6	84.9	85.2	85.5	85.8	86.1	86.4	86.7	87.0	87.3	87.6	87.9	88.2	88.5	88.7	89.0	89.3	89.5	89.8	90.1	90.4
6	82.9	83.2	83.4	83.7	84.0	84.3	84.6	84.9	85.2	85.5	85.8	86.1	86.4	86.7	87.0	87.3	87.6	87.9	88.2	88.5	88.7	89.0	89.3	89.5	89.8
7	82.4	82.7	82.9	83.2	83.5	83.8	84.1	84.4	84.7	85.0	85.3	85.6	85.9	86.2	86.5	86.8	87.1	87.4	87.7	88.0	88.3	88.6	88.9	89.2	89.5
8	81.9	82.2	82.4	82.7	83.0	83.3	83.6	83.9	84.2	84.5	84.8	85.1	85.4	85.7	86.0	86.3	86.6	86.9	87.2	87.5	87.8	88.1	88.4	88.7	89.0
9	81.4	81.7	81.9	82.2	82.5	82.8	83.1	83.4	83.7	84.0	84.3	84.6	84.9	85.2	85.5	85.8	86.1	86.4	86.7	87.0	87.3	87.6	87.9	88.2	88.5
Pol. =	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6	67.8	68.0	68.2	68.4	68.6	68.8	69.0	69.2	69.4	69.6	69.8
17.0	93.1	93.4	93.7	94.0	94.3	94.6	94.9	95.2	95.4	95.7	96.0	96.3	96.6	96.9	97.2	97.4	97.7	98.0	98.3	98.6	98.9	99.2	99.5	99.7	100.0
1	92.6	92.9	93.1	93.4	93.7	94.0	94.3	94.6	94.9	95.1	95.4	95.7	96.0	96.3	96.6	96.9	97.1	97.4	97.7	98.0	98.3	98.6	98.9	99.2	99.5
2	92.0	92.3	92.6	92.9	93.2	93.5	93.7	94.0	94.3	94.6	94.9	95.2	95.4	95.7	96.0	96.3	96.6	96.9	97.1	97.4	97.7	98.0	98.3	98.6	98.8
3	91.5	91.7	92.0	92.3	92.6	92.9	93.2	93.4	93.7	94.0	94.3	94.6	94.8	95.1	95.4	95.7	96.0	96.3	96.5	96.8	97.1	97.4	97.7	97.9	98.2
4	90.9	91.2	91.5	91.7	92.0	92.3	92.6	92.9	93.1	93.4	93.7	94.0	94.3	94.5	94.8	95.1	95.4	95.7	95.9	96.2	96.5	96.8	97.1	97.3	97.6
5	90.4	90.6	90.9	91.2	91.5	91.7	92.0	92.3	92.6	92.8	93.1	93.4	93.7	94.0	94.2	94.5	94.8	95.1	95.4	95.6	95.9	96.2	96.5	96.7	97.0
6	89.8	90.1	90.4	90.6	90.9	91.2	91.5	91.7	92.0	92.3	92.6	92.8	93.1	93.4	93.7	94.0	94.2	94.5	94.8	95.1	95.3	95.6	95.9	96.2	96.4
7	89.3	89.5	89.8	90.1	90.4	90.6	90.9	91.2	91.5	91.7	92.0	92.3	92.6	92.8	93.1	93.4	93.7	94.0	94.2	94.5	94.8	95.0	95.3	95.6	95.8
8	88.7	89.0	89.3	89.5	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	92.0	92.3	92.5	92.8	93.1	93.4	93.6	93.9	94.2	94.5	94.7	95.0	95.3
9	88.2	88.5	88.7	89.0	89.3	89.5	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	92.0	92.3	92.5	92.8	93.1	93.3	93.6	93.9	94.2	94.4	94.7
Pol. =	70.0	70.2	70.4	70.6	70.8	71.0	71.2	71.4	71.6	71.8	72.0	72.2	72.4	72.6	72.8	73.0	73.2	73.4	73.6	73.8	74.0	74.2	74.4	74.6	74.8
17.0	96.7	97.0	97.3	97.6	97.9	98.2	98.5	98.8	99.1	99.4	99.7	100.0													
1	96.1	96.4	96.7	97.0	97.3	97.6	97.9	98.2	98.5	98.8	99.1	99.4	99.7	100.0											
2	95.5	95.8	96.1	96.4	96.7	97.0	97.3	97.6	97.9	98.2	98.5	98.8	99.1	99.4	99.7	100.0									
3	94.9	95.2	95.5	95.8	96.1	96.4	96.7	97.0	97.3	97.6	97.9	98.2	98.5	98.8	99.1	99.4	99.7	100.0							
4	94.3	94.6	94.9	95.2	95.5	95.8	96.1	96.4	96.7	97.0	97.3	97.6	97.9	98.2	98.5	98.8	99.1	99.4	99.7	100.0					
5	93.7	94.0	94.3	94.6	94.9	95.2	95.5	95.8	96.1	96.4	96.7	97.0	97.3	97.6	97.9	98.2	98.5	98.8	99.1	99.4	99.7	100.0			
6	93.1	93.4	93.7	94.0	94.3	94.6	94.9	95.2	95.5	95.8	96.1	96.4	96.7	97.0	97.3	97.6	97.9	98.2	98.5	98.8	99.1	99.4	99.7	100.0	
7	92.5	92.8	93.1	93.4	93.7	94.0	94.3	94.6	94.9	95.2	95.5	95.8	96.1	96.4	96.7	97.0	97.3	97.6	97.9	98.2	98.5	98.8	99.1	99.4	99.7
8	91.9	92.2	92.5	92.8	93.1	93.4	93.7	94.0	94.3	94.6	94.9	95.2	95.5	95.8	96.1	96.4	96.7	97.0	97.3	97.6	97.9	98.2	98.5	98.8	99.1
9	91.3	91.6	91.9	92.2	92.5	92.8	93.1	93.4	93.7	94.0	94.3	94.6	94.9	95.2	95.5	95.8	96.1	96.4	96.7	97.0	97.3	97.6	97.9	98.2	98.5

TABLE 39—Continued  
EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	50.0	50.2	50.4	50.6	50.8	51.0	51.2	51.4	51.6	51.8	52.0	52.2	52.4	52.6	52.8	53.0	53.2	53.4	53.6	53.8	54.0	54.2	54.4	54.6	54.8
18.0	67.4	67.7	68.0	68.2	68.5	68.8	69.1	69.3	69.6	69.9	70.1	70.4	70.7	70.9	71.2	71.5	71.8	72.0	72.3	72.6	72.8	73.1	73.4	73.6	73.9
1	67.0	67.3	67.6	67.8	68.1	68.4	68.6	68.9	69.2	69.4	69.7	70.0	70.3	70.5	70.8	71.1	71.3	71.6	71.9	72.1	72.4	72.7	72.9	73.2	73.5
2	66.6	66.9	67.2	67.4	67.7	68.0	68.2	68.5	68.8	69.0	69.3	69.6	69.8	70.1	70.4	70.6	70.9	71.2	71.4	71.7	72.0	72.2	72.5	72.8	73.0
3	66.2	66.5	66.8	67.0	67.3	67.6	67.8	68.1	68.4	68.6	68.9	69.2	69.4	69.7	70.0	70.3	70.5	70.7	71.0	71.3	71.5	71.8	72.1	72.3	72.6
4	65.9	66.1	66.4	66.7	66.9	67.2	67.4	67.7	68.0	68.2	68.5	68.8	69.0	69.3	69.5	69.8	70.1	70.3	70.6	70.9	71.1	71.4	71.7	71.9	72.2
5	65.5	65.7	66.0	66.3	66.5	66.8	67.0	67.3	67.6	67.8	68.1	68.4	68.6	68.9	69.1	69.4	69.7	69.9	70.2	70.5	70.7	71.0	71.2	71.5	71.8
6	65.1	65.4	65.6	65.9	66.1	66.4	66.7	66.9	67.2	67.4	67.7	68.0	68.2	68.5	68.7	69.0	69.3	69.5	69.8	70.0	70.3	70.6	70.8	71.1	71.3
7	64.7	65.0	65.2	65.5	65.8	66.0	66.3	66.5	66.8	67.1	67.3	67.6	67.8	68.1	68.3	68.6	68.9	69.1	69.4	69.6	69.9	70.2	70.4	70.7	70.9
8	64.4	64.6	64.9	65.1	65.4	65.6	65.9	66.2	66.4	66.7	67.0	67.2	67.4	67.7	67.9	68.2	68.5	68.7	69.0	69.2	69.5	69.8	70.0	70.3	70.5
9	64.0	64.2	64.5	64.8	65.0	65.3	65.5	65.8	66.0	66.3	66.5	66.8	67.1	67.3	67.6	67.8	68.1	68.3	68.6	68.8	69.1	69.4	69.6	69.9	70.1
Pol. =	55.0	55.2	55.4	55.6	55.8	56.0	56.2	56.4	56.6	56.8	57.0	57.2	57.4	57.6	57.8	58.0	58.2	58.4	58.6	58.8	59.0	59.2	59.4	59.6	59.8
18.0	74.2	74.4	74.7	75.0	75.3	75.5	75.8	76.1	76.3	76.6	76.9	77.1	77.4	77.7	78.0	78.2	78.5	78.8	79.0	79.3	79.6	79.8	80.1	80.4	80.7
1	73.7	74.0	74.3	74.5	74.8	75.1	75.3	75.6	75.9	76.2	76.4	76.7	77.0	77.2	77.5	77.8	78.0	78.3	78.6	78.8	79.1	79.4	79.6	79.9	80.2
2	73.3	73.6	73.8	74.1	74.4	74.6	74.9	75.2	75.4	75.7	76.0	76.2	76.5	76.8	77.0	77.3	77.6	77.8	78.1	78.4	78.6	78.9	79.2	79.4	79.7
3	72.9	73.1	73.4	73.7	73.9	74.2	74.5	74.7	75.0	75.3	75.5	75.8	76.0	76.3	76.6	76.8	77.1	77.4	77.6	77.9	78.2	78.4	78.7	79.0	79.2
4	72.4	72.7	73.0	73.2	73.5	73.8	74.0	74.3	74.6	74.8	75.1	75.3	75.6	75.9	76.1	76.4	76.7	76.9	77.2	77.5	77.7	78.0	78.2	78.5	78.8
5	72.0	72.3	72.5	72.8	73.1	73.3	73.6	73.9	74.1	74.4	74.6	74.9	75.2	75.4	75.7	76.0	76.2	76.5	76.7	77.0	77.3	77.5	77.8	78.0	78.3
6	71.6	71.9	72.1	72.4	72.6	72.9	73.2	73.4	73.7	74.0	74.2	74.5	74.7	75.0	75.3	75.5	75.8	76.0	76.3	76.6	76.8	77.1	77.3	77.6	77.9
7	71.2	71.5	71.7	72.0	72.2	72.5	72.8	73.0	73.3	73.5	73.8	74.0	74.3	74.6	74.8	75.1	75.3	75.6	75.9	76.1	76.4	76.6	76.9	77.2	77.4
8	70.8	71.0	71.3	71.6	71.8	72.1	72.3	72.6	72.8	73.1	73.4	73.6	73.9	74.1	74.4	74.7	74.9	75.2	75.4	75.7	75.9	76.2	76.5	76.7	77.0
9	70.4	70.6	70.9	71.2	71.4	71.7	71.9	72.2	72.4	72.7	72.9	73.2	73.5	73.7	74.0	74.2	74.5	74.7	75.0	75.2	75.5	75.8	76.0	76.3	76.5
Pol. =	60.0	60.2	60.4	60.6	60.8	61.0	61.2	61.4	61.6	61.8	62.0	62.2	62.4	62.6	62.8	63.0	63.2	63.4	63.6	63.8	64.0	64.2	64.4	64.6	64.8
18.0	80.9	81.2	81.5	81.7	82.0	82.3	82.5	82.8	83.1	83.3	83.6	83.9	84.2	84.4	84.7	85.0	85.2	85.5	85.8	86.0	86.3	86.6	86.9	87.1	87.4
1	80.4	80.7	81.0	81.2	81.5	81.8	82.1	82.3	82.6	82.9	83.1	83.4	83.7	83.9	84.2	84.5	84.7	85.0	85.3	85.6	85.8	86.1	86.3	86.6	86.9
2	80.0	80.2	80.5	80.8	81.0	81.3	81.6	81.8	82.1	82.4	82.6	82.9	83.2	83.4	83.7	84.0	84.2	84.5	84.8	85.0	85.3	85.6	85.8	86.1	86.4
3	79.5	79.8	80.0	80.3	80.6	80.8	81.1	81.3	81.6	81.9	82.1	82.4	82.7	82.9	83.2	83.5	83.7	84.0	84.3	84.5	84.8	85.1	85.3	85.6	85.9
4	79.0	79.3	79.6	79.8	80.1	80.3	80.6	80.9	81.1	81.4	81.7	81.9	82.2	82.5	82.7	83.0	83.2	83.5	83.8	84.0	84.3	84.6	84.8	85.1	85.4
5	78.6	78.8	79.1	79.4	79.6	79.9	80.1	80.4	80.7	80.9	81.3	81.5	81.7	82.0	82.2	82.5	82.8	83.0	83.3	83.5	83.8	84.1	84.3	84.6	84.8
6	78.1	78.4	78.6	78.9	79.2	79.4	79.7	79.9	80.2	80.5	80.7	81.0	81.2	81.5	81.8	82.0	82.3	82.5	82.8	83.1	83.3	83.6	83.8	84.1	84.4
7	77.7	77.9	78.2	78.5	78.8	79.0	79.3	79.5	79.7	79.9	80.3	80.6	80.8	81.0	81.3	81.6	81.8	82.1	82.3	82.6	82.8	83.1	83.4	83.6	83.9
8	77.2	77.5	77.7	78.0	78.3	78.5	78.8	79.0	79.3	79.5	79.8	80.1	80.3	80.6	80.8	81.1	81.3	81.6	81.9	82.1	82.4	82.6	82.9	83.1	83.4
9	76.8	77.0	77.3	77.5	77.8	78.1	78.3	78.6	78.8	79.1	79.3	79.6	79.9	80.1	80.4	80.6	80.9	81.1	81.4	81.6	81.9	82.2	82.4	82.7	82.9



TABLE 39—Continued  
EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	65.0	66.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6	67.8	68.0	68.2	68.4	68.6	68.8	69.0	69.2	69.4	69.6	69.8
18.0	87.7	87.9	88.2	88.5	88.7	89.0	89.3	89.6	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	92.0	92.3	92.5	92.8	93.1	93.3	93.6	93.9	94.1
.1	87.1	87.4	87.7	87.9	88.2	88.5	88.8	89.0	89.3	89.6	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	92.0	92.2	92.5	92.8	93.0	93.3	93.6
.2	86.6	83.9	87.2	87.4	87.7	88.0	88.2	88.5	88.8	89.0	89.3	89.6	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	92.0	92.2	92.5	92.8	93.0
.3	86.1	83.4	86.6	86.9	87.2	87.4	87.7	88.0	88.2	88.5	88.8	89.0	89.3	89.6	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	91.9	92.2	92.5
.4	85.6	85.9	86.1	86.4	86.7	86.9	87.2	87.5	87.7	88.0	88.3	88.5	88.8	89.0	89.3	89.6	89.8	90.1	90.4	90.6	90.9	91.2	91.4	91.7	91.9
.5	85.1	85.4	85.6	85.9	86.2	86.4	86.7	87.0	87.2	87.5	87.7	88.0	88.3	88.5	88.8	89.0	89.3	89.6	90.0	90.3	90.6	90.9	91.1	91.4	91.6
.6	84.6	84.9	85.2	85.4	85.7	85.9	86.2	86.5	86.7	87.0	87.2	87.5	87.8	88.0	88.3	88.5	88.8	89.0	89.3	89.6	90.0	90.3	90.6	90.9	91.1
.7	84.1	84.4	84.7	84.9	85.2	85.4	85.7	86.0	86.2	86.5	86.7	87.0	87.2	87.5	87.8	88.0	88.3	88.5	88.8	89.0	89.3	89.6	89.8	90.1	90.4
.8	83.7	83.9	84.2	84.4	84.7	84.9	85.2	85.5	85.7	86.0	86.2	86.5	86.7	87.0	87.2	87.5	87.8	88.0	88.3	88.5	88.8	89.1	89.3	89.6	89.8
.9	83.2	83.4	83.7	83.9	84.2	84.5	84.7	85.0	85.2	85.5	85.7	86.0	86.3	86.5	86.8	87.0	87.3	87.5	87.8	88.0	88.3	88.5	88.8	89.1	89.3
Pol. =	70.0	70.2	70.4	70.6	70.8	71.0	71.2	71.4	71.6	71.8	72.0	72.2	72.4	72.6	72.8	73.0	73.2	73.4	73.6	73.8	74.0	74.2	74.4	74.6	74.8
18.0	94.4	94.7	94.9	95.2	95.5	95.8	96.0	96.3	96.6	96.8	97.1	97.4	97.6	97.9	98.2	98.5	98.7	99.0	99.3	99.5	99.8	100.1	99.7	100.0	99.7
.1	93.8	94.1	94.4	94.7	94.9	95.2	95.5	95.7	96.0	96.3	96.5	96.8	97.1	97.3	97.6	97.9	98.1	98.4	98.7	98.9	99.2	99.5	99.7	100.0	99.7
.2	93.3	93.6	93.8	94.1	94.4	94.6	94.9	95.2	95.4	95.7	96.0	96.2	96.5	96.8	97.0	97.3	97.6	97.8	98.1	98.3	98.6	98.9	99.2	99.4	99.7
.3	92.7	93.0	93.3	93.5	93.8	94.1	94.3	94.6	94.9	95.1	95.4	95.7	95.9	96.2	96.5	96.7	97.0	97.2	97.5	97.8	98.0	98.3	98.6	98.8	99.1
.4	92.2	92.5	92.7	93.0	93.3	93.5	93.8	94.0	94.3	94.6	94.8	95.1	95.4	95.6	95.9	96.2	96.4	96.7	96.9	97.2	97.5	97.7	98.0	98.3	98.5
.5	91.7	91.9	92.2	92.5	92.7	93.0	93.2	93.5	93.8	94.0	94.3	94.5	94.8	95.1	95.3	95.6	95.9	96.1	96.4	96.6	96.9	97.2	97.4	97.7	98.0
.6	91.1	91.4	91.7	91.9	92.2	92.4	92.7	93.0	93.2	93.5	93.7	94.0	94.3	94.5	94.8	95.0	95.3	95.6	95.8	96.1	96.3	96.6	96.9	97.1	97.4
.7	90.6	90.9	91.1	91.4	91.7	91.9	92.2	92.4	92.7	92.9	93.2	93.5	93.7	94.0	94.3	94.5	94.8	95.0	95.3	95.5	95.8	96.1	96.3	96.6	96.8
.8	90.1	90.4	90.6	90.9	91.1	91.4	91.6	91.9	92.2	92.4	92.7	92.9	93.2	93.4	93.7	94.0	94.2	94.5	94.7	95.0	95.2	95.5	95.8	96.0	96.3
.9	89.6	89.8	90.1	90.3	90.6	90.9	91.1	91.4	91.6	91.9	92.1	92.4	92.7	92.9	93.2	93.4	93.7	93.9	94.2	94.4	94.7	95.0	95.2	95.5	95.7
Pol. =	75.0	75.2	75.4	75.6	75.8	76.0	76.2	76.4	76.6	76.8	77.0	77.2	77.4	77.6	77.8	78.0	78.2	78.4	78.6	78.8	79.0	79.2	79.4	79.6	79.8
18.0																									
.1	100.0																								
.2	99.4	99.6	99.9	100.2																					
.3	98.8	99.1	99.3	99.6	99.8	100.1																			
.4	98.2	98.5	98.7	99.0	99.3	99.5	99.8	100.0																	
.5	97.7	97.9	98.2	98.4	98.7	99.0	99.2	99.5	99.7	100.0															
.6	97.1	97.3	97.6	97.9	98.1	98.4	98.6	98.9	99.2	99.4	99.7	99.9	100.2												
.7	96.5	96.8	97.0	97.3	97.6	97.8	98.1	98.3	98.6	98.8	99.1	99.4	99.6	99.9	100.1										
.8	96.0	96.2	96.5	96.7	97.0	97.3	97.5	97.8	98.0	98.3	98.5	98.8	99.0	99.3	99.6	99.8	100.1								
.9	95.5	95.7	96.0	96.3	96.5	96.8	97.0	97.3	97.5	97.8	98.0	98.3	98.5	98.8	99.0	99.3	99.6	99.8	100.1						

EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	55.0	55.2	55.4	55.6	55.8	56.0	56.2	56.4	56.6	56.8	57.0	57.2	57.4	57.6	57.8	58.0	58.2	58.4	58.6	58.8	59.0	59.2	59.4	59.6	59.8
19.0	70.0	70.2	70.5	70.8	71.0	71.3	71.5	71.8	72.0	72.3	72.5	72.8	73.0	73.3	73.6	73.8	74.1	74.3	74.6	74.8	75.1	75.3	75.6	75.8	76.1
.1	69.6	69.8	70.1	70.4	70.6	70.9	71.1	71.4	71.6	71.9	72.1	72.3	72.6	72.8	73.1	73.3	73.6	73.8	74.1	74.3	74.6	74.8	75.1	75.3	75.6
.2	69.2	69.5	69.7	70.0	70.2	70.5	70.7	71.0	71.2	71.5	71.7	72.0	72.2	72.5	72.7	73.0	73.2	73.5	73.7	74.0	74.2	74.5	74.7	75.0	75.2
.3	68.8	69.1	69.3	69.6	69.8	70.1	70.3	70.6	70.8	71.1	71.3	71.6	71.8	72.1	72.3	72.6	72.8	73.1	73.3	73.6	73.8	74.1	74.3	74.6	74.8
.4	68.4	68.7	68.9	69.2	69.4	69.7	69.9	70.2	70.4	70.7	70.9	71.1	71.4	71.7	71.9	72.2	72.4	72.7	72.9	73.2	73.4	73.7	73.9	74.2	74.4
.5	68.1	68.3	68.5	68.8	69.0	69.3	69.5	69.8	70.0	70.3	70.5	70.8	71.0	71.3	71.5	71.8	72.0	72.3	72.5	72.8	73.0	73.2	73.5	73.7	74.0
.6	67.7	67.9	68.2	68.4	68.7	68.9	69.2	69.4	69.6	69.9	70.1	70.4	70.6	70.9	71.1	71.4	71.6	71.9	72.1	72.4	72.6	72.8	73.1	73.3	73.6
.7	67.3	67.5	67.8	68.0	68.3	68.5	68.8	69.0	69.3	69.5	69.8	70.0	70.3	70.5	70.7	71.0	71.2	71.5	71.7	72.0	72.2	72.4	72.7	72.9	73.2
.8	66.9	67.2	67.4	67.7	67.9	68.2	68.4	68.6	68.9	69.1	69.4	69.6	69.9	70.1	70.3	70.6	70.8	71.1	71.3	71.6	71.8	72.1	72.3	72.5	72.8
.9	66.6	66.8	67.1	67.3	67.5	67.8	68.0	68.3	68.5	68.8	69.0	69.2	69.5	69.7	70.0	70.2	70.4	70.7	70.9	71.2	71.4	71.7	71.9	72.1	72.4
Pol. =	60.0	60.2	60.4	60.6	60.8	61.0	61.2	61.4	61.6	61.8	62.0	62.2	62.4	62.6	62.8	63.0	63.2	63.4	63.6	63.8	64.0	64.2	64.4	64.6	64.8
19.0	76.4	76.6	76.9	77.1	77.4	77.6	77.9	78.1	78.4	78.6	78.9	79.1	79.4	79.7	79.9	80.2	80.4	80.7	80.9	81.2	81.4	81.7	81.9	82.2	82.5
.1	75.9	76.2	76.4	76.7	76.9	77.2	77.4	77.7	77.9	78.2	78.4	78.7	79.0	79.2	79.5	79.7	80.0	80.2	80.5	80.7	81.0	81.2	81.5	81.7	82.0
.2	75.5	75.7	76.0	76.2	76.5	76.8	77.0	77.3	77.5	77.8	78.0	78.3	78.5	78.8	79.0	79.3	79.5	79.8	80.0	80.3	80.5	80.8	81.0	81.3	81.5
.3	75.1	75.3	75.6	75.8	76.1	76.3	76.6	76.8	77.1	77.3	77.6	77.8	78.1	78.3	78.6	78.8	79.1	79.3	79.6	79.8	80.1	80.3	80.6	80.8	81.1
.4	74.7	74.9	75.1	75.4	75.6	75.9	76.1	76.4	76.6	76.9	77.1	77.4	77.6	77.9	78.1	78.4	78.6	78.9	79.1	79.4	79.6	79.9	80.1	80.4	80.6
.5	74.2	74.5	74.7	75.0	75.3	75.5	75.7	76.0	76.2	76.5	76.7	77.0	77.2	77.5	77.7	77.9	78.2	78.4	78.7	78.9	79.2	79.4	79.7	79.9	80.2
.6	73.8	74.1	74.3	74.6	74.8	75.1	75.3	75.6	75.8	76.0	76.3	76.5	76.8	77.0	77.3	77.5	77.8	78.0	78.3	78.5	78.8	79.0	79.2	79.5	79.7
.7	73.4	73.7	73.9	74.2	74.4	74.6	74.9	75.1	75.4	75.6	75.9	76.1	76.4	76.6	76.8	77.1	77.3	77.6	77.8	78.1	78.3	78.6	78.8	79.1	79.3
.8	73.0	73.3	73.5	73.8	74.0	74.2	74.5	74.7	75.0	75.2	75.5	75.7	75.9	76.2	76.4	76.7	76.9	77.2	77.4	77.7	77.9	78.1	78.4	78.6	78.9
.9	72.6	72.9	73.1	73.4	73.6	73.8	74.1	74.3	74.6	74.8	75.0	75.3	75.5	75.8	76.0	76.3	76.5	76.7	77.0	77.2	77.5	77.7	77.9	78.2	78.4
Pol. =	65.0	65.2	65.4	65.6	65.8	66.0	66.2	66.4	66.6	66.8	67.0	67.2	67.4	67.6	67.8	68.0	68.2	68.4	68.6	68.8	69.0	69.2	69.4	69.6	69.8
19.0	82.7	83.0	83.2	83.5	83.7	84.0	84.2	84.5	84.7	85.0	85.3	85.5	85.8	86.0	86.3	86.5	86.8	87.0	87.3	87.5	87.8	88.1	88.3	88.6	88.8
.1	82.2	82.5	82.8	83.0	83.3	83.5	83.8	84.0	84.3	84.5	84.8	85.0	85.3	85.5	85.8	86.0	86.3	86.5	86.8	87.1	87.3	87.6	87.8	88.1	88.3
.2	81.8	82.0	82.3	82.5	82.8	83.0	83.3	83.5	83.8	84.0	84.3	84.5	84.8	85.1	85.3	85.6	85.8	86.1	86.3	86.6	86.8	87.1	87.3	87.6	87.8
.3	81.3	81.6	81.8	82.1	82.3	82.6	82.8	83.1	83.3	83.6	83.8	84.1	84.3	84.6	84.8	85.1	85.3	85.6	85.8	86.1	86.3	86.6	86.8	87.1	87.3
.4	80.9	81.1	81.4	81.6	81.9	82.1	82.4	82.6	82.9	83.1	83.3	83.6	83.9	84.1	84.4	84.6	84.9	85.1	85.4	85.6	85.9	86.1	86.3	86.6	86.8
.5	80.4	80.7	80.9	81.2	81.4	81.7	81.9	82.2	82.4	82.7	82.9	83.1	83.4	83.6	83.9	84.1	84.4	84.6	84.9	85.1	85.4	85.6	85.9	86.1	86.4
.6	79.8	80.2	80.5	80.7	81.0	81.2	81.5	81.7	82.0	82.2	82.4	82.7	82.9	83.2	83.4	83.7	83.9	84.2	84.4	84.7	84.9	85.2	85.4	85.6	85.9
.7	79.5	79.8	80.0	80.3	80.5	80.8	81.0	81.3	81.5	81.7	82.0	82.2	82.5	82.7	83.0	83.2	83.5	83.7	83.9	84.2	84.4	84.7	84.9	85.2	85.4
.8	79.1	79.4	79.6	79.8	80.1	80.3	80.6	80.8	81.1	81.3	81.5	81.8	82.0	82.3	82.5	82.8	83.0	83.3	83.5	83.7	84.0	84.2	84.5	84.7	85.0
.9	78.7	78.9	79.2	79.4	79.6	79.9	80.1	80.4	80.6	80.9	81.1	81.3	81.6	81.8	82.1	82.3	82.5	82.8	83.0	83.3	83.5	83.8	84.0	84.2	84.5

TABLE 39—Continued

Pol. =	70.0	70.2	70.4	70.6	70.8	71.0	71.2	71.4	71.6	71.8	72.0	72.2	72.4	72.6	72.8	73.0	73.2	73.4	73.6	73.8	74.0	74.2	74.4	74.6	74.8
19.0	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.9	91.1	91.4	91.6	91.9	92.1	92.4	92.6	92.9	93.1	93.4	93.7	93.9	94.2	94.4	94.7	94.9	95.2
.1	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.6	91.9	92.1	92.4	92.6	92.9	93.1	93.4	93.6	93.9	94.1	94.4	94.6
.2	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.6	91.8	92.1	92.3	92.6	92.8	93.1	93.4	93.6	93.9	94.1
.3	87.6	87.8	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.6	91.8	92.1	92.3	92.6	92.8	93.1	93.3	93.6
.4	87.1	87.3	87.6	87.8	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.6	91.8	92.1	92.3	92.6	92.8	93.1
.5	86.6	86.9	87.1	87.4	87.6	87.8	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.6	91.8	92.1	92.3	92.6
.6	86.1	86.4	86.6	86.9	87.1	87.4	87.6	87.8	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.6	91.8	92.1
.7	85.7	85.9	86.1	86.4	86.6	86.9	87.1	87.4	87.6	87.8	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.1	91.3	91.6
.8	85.2	85.4	85.7	85.9	86.2	86.4	86.7	86.9	87.1	87.4	87.6	87.8	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.6	90.8	91.0
.9	84.7	85.0	85.2	85.5	85.7	85.9	86.2	86.4	86.7	86.9	87.1	87.4	87.6	87.8	88.1	88.3	88.6	88.8	89.1	89.3	89.6	89.8	90.1	90.3	90.5
Pol. =	75.0	75.2	75.4	75.6	75.8	76.0	76.2	76.4	76.6	76.8	77.0	77.2	77.4	77.6	77.8	78.0	78.2	78.4	78.6	78.8	79.0	79.2	79.4	79.6	79.8
19.0	95.4	95.7	95.9	96.2	96.5	96.7	97.0	97.2	97.5	97.7	98.0	98.2	98.5	98.7	99.0	99.3	99.5	99.8	100.0						
.1	94.9	95.2	95.4	95.7	95.9	96.2	96.4	96.7	96.9	97.2	97.4	97.7	97.9	98.2	98.4	98.7	98.9	99.2	99.5	99.7	100.0				
.2	94.4	94.6	94.9	95.1	95.4	95.6	95.9	96.1	96.4	96.6	96.9	97.1	97.4	97.6	97.9	98.1	98.4	98.6	98.9	99.1	99.4	99.6	99.9	100.2	
.3	93.8	94.1	94.3	94.6	94.8	95.1	95.3	95.6	95.8	96.1	96.3	96.6	96.8	97.1	97.3	97.6	97.8	98.1	98.3	98.6	98.8	99.1	99.3	99.6	99.8
.4	93.3	93.6	93.8	94.1	94.3	94.6	94.8	95.1	95.3	95.6	95.8	96.1	96.3	96.6	96.8	97.0	97.3	97.5	97.8	98.0	98.3	98.5	98.8	99.0	99.3
.5	92.8	93.1	93.3	93.6	93.8	94.0	94.3	94.5	94.8	94.9	95.3	95.5	95.8	96.0	96.3	96.5	96.8	97.0	97.3	97.5	97.7	98.0	98.2	98.5	98.7
.6	92.3	92.5	92.8	93.0	93.3	93.5	93.8	94.0	94.3	94.5	94.7	95.0	95.2	95.5	95.7	96.0	96.2	96.5	96.7	97.0	97.2	97.5	97.7	97.9	98.2
.7	91.8	92.0	92.3	92.5	92.8	93.0	93.2	93.5	93.7	94.0	94.2	94.5	94.7	95.0	95.2	95.4	95.7	95.9	96.2	96.4	96.7	96.9	97.2	97.4	97.7
.8	91.3	91.5	91.8	92.0	92.3	92.5	92.7	93.0	93.2	93.5	93.7	94.0	94.2	94.4	94.7	94.9	95.2	95.4	95.7	95.9	96.2	96.4	96.6	96.9	97.1
.9	90.8	91.0	91.3	91.5	91.7	92.0	92.2	92.5	92.7	93.0	93.2	93.4	93.7	93.9	94.2	94.4	94.7	94.9	95.1	95.4	95.6	95.9	96.1	96.3	96.6
Pol. =	80.0	80.2	80.4	80.6	80.8	81.0	81.2	81.4	81.6	81.8	82.0	82.2	82.4	84.6	84.8	83.0	83.2	83.4	83.6	83.8	84.0	84.2	84.4	84.6	84.8
19.0																									
.1																									
.2																									
.3	100.1																								
.4	99.5	99.8	100.0																						
.5	99.0	99.2	99.5	99.7	100.0																				
.6	98.4	98.7	98.9	99.2	99.4																				
.7	97.9	98.1	98.4	98.6	98.9	99.1																			
.8	97.4	97.6	97.9	98.1	98.3	98.6	98.8	99.1																	
.9	96.8	97.1	97.3	97.6	97.8	98.0	98.3	98.5	98.8	99.0	99.3	99.5	99.7	100.0											

TABLE 39—Continued  
EXPANDED HORNE'S TABLE OF COEFFICIENTS OF PURITY

Pol. =	60.0	60.5	61.0	61.5	62.0	62.5	63.0	63.5	64.0	64.5	65.0
20.0	72.2	72.5	72.7	73.0	73.2	73.4	73.7	73.9	74.2	74.4	74.6
.1	71.8	72.1	72.3	72.6	72.8	73.0	73.3	73.5	73.8	74.0	74.2
.2	71.5	71.7	71.9	72.2	72.4	72.7	72.9	73.1	73.4	73.6	73.8
.3	71.1	71.3	71.5	71.8	72.0	72.3	72.5	72.7	73.0	73.2	73.4
.4	70.7	70.9	71.2	71.4	71.6	71.9	72.1	72.4	72.6	72.8	73.1
.5	70.3	70.6	70.8	71.0	71.3	71.5	71.7	72.0	72.2	72.4	72.7
.6	70.0	70.2	70.4	70.7	71.0	71.2	71.4	71.6	71.9	72.1	72.3
.7	69.6	69.8	70.1	70.3	70.5	70.7	71.0	71.2	71.4	71.6	71.9
.8	69.2	69.5	69.7	69.9	70.2	70.4	70.6	70.8	71.1	71.3	71.5
.9	68.9	69.1	69.3	69.6	69.8	70.0	70.2	70.5	70.7	70.9	71.2
Pol. =	65.0	65.5	66.0	66.5	67.0	67.5	68.0	68.5	69.0	69.5	70.0
20.0	78.3	78.5	78.7	79.0	79.2	79.5	79.7	79.9	80.2	80.4	80.7
.1	77.8	78.1	78.3	78.6	78.8	79.1	79.3	79.6	79.8	80.0	80.2
.2	77.4	77.7	77.9	78.1	78.4	78.7	78.9	79.1	79.4	79.6	79.8
.3	77.0	77.2	77.5	77.7	78.0	78.2	78.4	78.7	78.9	79.1	79.3
.4	76.6	76.8	77.1	77.3	77.5	77.8	78.0	78.2	78.5	78.7	78.9
.5	76.2	76.4	76.7	76.9	77.1	77.4	77.6	77.8	78.1	78.3	78.5
.6	75.8	76.0	76.3	76.5	76.7	77.0	77.2	77.5	77.7	77.9	78.1
.7	75.4	75.6	75.9	76.1	76.3	76.6	76.8	77.0	77.3	77.5	77.8
.8	75.0	75.2	75.5	75.7	75.9	76.2	76.4	76.6	76.9	77.1	77.3
.9	74.6	74.8	75.0	75.2	75.4	75.6	75.8	76.0	76.2	76.4	76.6
Pol. =	70.0	70.5	71.0	71.5	72.0	72.5	73.0	73.5	74.0	74.5	75.0
20.0	84.3	84.5	84.8	85.0	85.2	85.5	85.7	86.0	86.2	86.4	86.7
.1	83.8	84.1	84.3	84.5	84.8	85.0	85.3	85.5	85.7	86.0	86.2
.2	83.4	83.6	83.8	84.1	84.3	84.6	84.8	85.1	85.3	85.5	85.8
.3	82.9	83.1	83.3	83.6	83.8	84.1	84.3	84.6	84.8	85.1	85.3
.4	82.5	82.7	82.9	83.2	83.4	83.7	83.9	84.1	84.4	84.6	84.8
.5	82.0	82.3	82.5	82.8	83.0	83.3	83.5	83.7	84.0	84.2	84.4
.6	81.6	81.9	82.1	82.4	82.6	82.8	83.0	83.3	83.5	83.7	84.0
.7	81.2	81.4	81.6	81.9	82.1	82.3	82.6	82.8	83.0	83.3	83.5
.8	80.8	81.0	81.2	81.5	81.7	81.9	82.2	82.4	82.6	82.8	83.1
.9	80.3	80.6	80.8	81.0	81.3	81.5	81.7	82.0	82.2	82.4	82.6



TABLE 40  
EXPANDED MEISSEL-HILDE TABLE FOR INVERT SUGAR.\*—E. W. RICE

Weight of Sample in 100 ML.		1 Gram		2 Grams		5 Grams		10 Grams		20 Grams	
		30°	35°	20°	30°	85°	95°	85°	95°	85°	95°
Polarization											
Weight Obtained as											
Cu	Cu <sub>2</sub> O	CuO									
.0999	.1125	.1250	10.28	10.26	5.13	5.12	1.661	1.600	.76	.76	
.1019	.1147	.1275	10.48	10.46	5.24	5.23	1.708	1.648	.78	.78	
.1039	.1170	.1300	10.69	10.67	5.34	5.33	1.765	1.696	.80	.80	
.1059	.1192	.1325	10.89	10.87	5.44	5.43	1.802	1.744	.82	.82	
.1070	.1215	.1350	11.10	11.08	5.54	5.53	1.849	1.792	.84	.84	
.1090	.1237	.1375	11.31	11.29	5.65	5.63	1.896	1.839	.86	.86	
.1110	.1260	.1400	11.52	11.50	5.76	5.74	1.942	1.886	.88	.88	
.1138	.1282	.1425	11.73	11.71	5.86	5.84	1.989	1.933	.90	.90	
.1158	.1305	.1450	11.94	11.92	5.96	5.95	2.038	1.980	.92	.92	
.1178	.1327	.1475	12.15	12.13	6.07	6.05	2.082	2.027	.94	.94	
.1198	.1350	.1500	12.36	12.34	6.18	6.16	2.128	2.074	.96	.96	
.1218	.1372	.1525	12.57	12.55	6.28	6.26	2.175	2.121	.98	.98	
.1238	.1395	.1550	12.78	12.76	6.38	6.36	2.221	2.168	1.00	1.00	
.1258	.1417	.1575	12.99	12.97	6.49	6.47	2.267	2.215	1.02	1.02	
.1278	.1440	.1600	13.21	13.19	6.60	6.58	2.313	2.262	1.05	1.04	
.1298	.1462	.1625	13.42	13.40	6.70	6.68	2.359	2.309	1.07	1.06	
.1318	.1485	.1650	13.64	13.62	6.81	6.79	2.405	2.358	1.09	1.08	
.1338	.1507	.1675	13.85	13.83	6.92	6.90	2.451	2.403	1.11	1.10	
.1358	.1530	.1700	14.07	14.05	7.03	7.01	2.497	2.449	1.13	1.12	
.1378	.1552	.1725	14.28	14.26	7.13	7.11	2.543	2.496	1.15	1.14	
.1398	.1575	.1750	14.50	14.48	7.24	7.22	2.589	2.543	1.17	1.16	
.1418	.1597	.1775	14.72	14.69	7.35	7.33	2.635	2.589	1.19	1.18	
.1438	.1620	.1800	14.93	14.91	7.46	7.44	2.680	2.635	1.22	1.21	

\* 8th Int. Cong. App. Chemistry, 8, 47.

NOTE.—The fourth and fifth pages of this table have been extended to include concentrations of 1.5 grams per 100 ml. (See method for glucose in molasses, page 328.)

TABLE 40—Continued  
EXPANDED MEISSL-HILLER TABLE FOR INVERT SUGAR—E. W. RICE

Weight of Sample in 100 MI.			1 Gram		2 Grams		5 Grams		10 Grams		20 Grams	
Polarization			30°	35°	20°	30°	85°	95°	85°	95°	85°	95°
			Weight Obtained as									
Cu	Cu <sub>2</sub> O	CuO										
.1458	.1642	.1825	15.15	15.12	7.56	7.54	2.726	2.682	1.24	1.23		
.1478	.1665	.1850	15.37	15.43	7.67	7.65	2.772	2.728	1.27	1.25		
.1498	.1687	.1875	15.59	15.56	7.78	7.76	2.817	2.774	1.29	1.27		
.1518	.1710	.1900	15.81	15.78	7.89	7.87	2.862	2.820	1.31	1.30		
.1538	.1732	.1925	16.03	16.00	7.99	7.97	2.907	2.867	1.33	1.32		
.1558	.1755	.1950	16.25	16.22	8.10	8.08	2.952	2.913	1.35	1.34		
.1578	.1777	.1975	16.47	16.44	8.21	8.19	2.997	2.959	1.37	1.36		
.1598	.1800	.2000	16.69	16.66	8.32	8.30	3.042	3.005	1.40	1.38		
.1618	.1822	.2025	16.91	16.88	8.43	8.40	3.087	3.051	1.42	1.40		
.1638	.1845	.2050	17.13	17.10	8.54	8.51	3.132	3.097	1.44	1.42		
.1658	.1867	.2075	17.35	17.32	8.65	8.62	3.177	3.143	1.46	1.44		
.1678	.1890	.2100	17.57	17.54	8.76	8.73	3.221	3.188	1.49	1.47		
.1698	.1912	.2125	17.79	17.76	8.87	8.83	3.266	3.234	1.51	1.49		
.1718	.1935	.2150	18.01	17.98	8.98	8.94	3.310	3.280	1.53	1.51		
.1738	.1957	.2175	18.23	18.20	9.09	9.05	3.354	3.325	1.55	1.53	.72	
.1758	.1980	.2200	18.45	18.42	9.20	9.16	3.398	3.370	1.58	1.56	.73	
.1778	.2002	.2225	18.67	18.64	9.31	9.26	3.433	3.416	1.60	1.58	.75	
.1798	.2025	.2250	18.89	18.86	9.42	9.37	3.488	3.461	1.62	1.60	.77	
.1817	.2047	.2275	19.11	19.08	9.53	9.48	3.532	3.506	1.64	1.62	.78	
.1837	.2070	.2300	19.34	19.30	9.64	9.59	3.576	3.551	1.67	1.65	.79	
.1857	.2092	.2325	19.56	19.52	9.75	9.70	3.621	3.597	1.69	1.67	.80	
.1877	.2115	.2350	19.78	19.74	9.86	9.81	3.666	3.642	1.71	1.69	.81	
.1897	.2137	.2375	20.00	19.96	9.97	9.92	3.710	3.687	1.73	1.72	.82	.81
.1917	.2160	.2400	20.23	20.19	10.08	10.03	3.754	3.732	1.76	1.75	.83	.83
.1937	.2182	.2425	20.45	20.41	10.19	10.14	3.799	3.777	1.78	1.77	.84	.84
.1957	.2205	.2450	20.67	20.63	10.30	10.25	3.844	3.822	1.80	1.79	.85	.85





TABLE 40—Continued  
EXPANDED MEISSL-HILLER TABLE FOR INVERT SUGAR.—E. W. RICE

Wt. of Sample in 100 ML.	1 Gram		1.5 Grams		2 Grams		5 Grams		10 Grams		20 Grams	
	30°	35°	25°	35°	20°	30°	85°	95°	85°	95°	85°	95°
Polarization												
Wt. Obtained as												
	Cu	CuO										
.2497	3125	26.67	17.71	17.64	13.29	13.22	5.046	5.031	2.44	2.42	1.16	1.15
.2517	3150	26.90	17.85	17.79	13.40	13.33	5.095	5.076	2.46	2.44	1.17	1.15
.2537	3175	27.12	18.01	17.94	13.51	13.44	5.141	5.121	2.48	2.46	1.18	1.17
.2556	3200	27.35	18.16	18.08	13.63	13.56	5.186	5.166	2.51	2.49	1.19	1.18
.2576	3225	27.57	18.31	18.23	13.74	13.67	5.232	5.212	2.53	2.51	1.20	1.19
.2596	3250	27.79	18.46	18.38	13.85	13.78	5.278	5.258	2.55	2.53	1.21	1.20
.2616	3275	28.02	18.62	18.54	13.96	13.89	5.324	5.303	2.57	2.55	1.22	1.21
.2636	3300	28.25	18.77	18.69	14.08	14.01	5.371	5.348	2.60	2.58	1.24	1.23
.2656	3325	28.47	18.92	18.84	14.19	14.12	5.418	5.394	2.62	2.60	1.25	1.24
.2676	3350	28.69	19.07	18.99	14.30	14.23	5.465	5.440	2.64	2.62	1.26	1.25
.2696	3375	28.87	19.22	19.14	14.42	14.35	5.512	5.486	2.66	2.64	1.27	1.26
.2716	3400	29.10	19.38	19.30	14.54	14.47	5.558	5.532	2.69	2.67	1.28	1.27
.2736	3425	29.32	19.53	19.45	14.65	14.59	5.605	5.578	2.71	2.69	1.29	1.28
.2756	3450	29.55	19.69	19.61	14.76	14.70	5.652	5.624	2.73	2.71	1.30	1.29
.2776	3475	29.77	19.85	19.77	14.88	14.81	5.699	5.671	2.75	2.73	1.31	1.30
.2796	3500	30.00	20.00	19.92	15.00	14.93	5.746	5.718	2.78	2.76	1.33	1.32
.2816	3525	30.31	20.16	20.08	15.11	15.04	5.793	5.765	2.80	2.78	1.34	1.33
.2836	3550	30.54	20.31	20.23	15.22	15.15	5.840	5.812	2.82	2.80	1.35	1.34
.2856	3575	30.77	20.47	20.39	15.34	15.27	5.888	5.859	2.84	2.82	1.36	1.35
.2876	3600	31.00	20.61	20.53	15.46	15.39	5.936	5.906	2.87	2.85	1.37	1.36
.2896	3625	31.23	20.76	20.68	15.57	15.50	5.983	5.953	2.89	2.87	1.38	1.37
.2916	3650	31.46	20.92	20.84	15.69	15.61	6.031	6.000	2.91	2.89	1.39	1.38
.2936	3675	31.69	21.07	20.99	15.81	15.73	6.079	6.048	2.93	2.91	1.40	1.39
.2956	3700	31.93	21.23	21.15	15.93	15.85	6.127	6.096	2.96	2.94	1.42	1.41
.2976	3725	32.16	21.38	21.30	16.04	15.96	6.174	6.144	2.98	2.96	1.43	1.42
.2996	3750	32.40	21.53	21.45	16.16	16.08	6.222	6.192	3.00	2.98	1.44	1.43

TABLE 40—Continued  
EXPANDED MEISSNER-HILLER TABLE FOR INVERT SUGAR.—E. W. RICE

Wt. of Sample in 100 ML.	1 Gram		1.5 Grams		2 Grams		5 Grams		10 Grams		20 Grams	
	30°	35°	25°	35°	20°	30°	85°	95°	85°	95°	85°	95°
Polarization												
Wt. Obtained as												
Cu												
CuO												
.3016	32.63	32.57	21.69	21.61	16.28	16.20	6.270	6.240	3.03	3.00	1.45	1.44
.3036	32.87	32.81	21.86	21.77	16.40	16.32	6.318	6.288	3.06	3.03	1.46	1.45
.3056	33.10	33.04	22.00	21.93	16.52	16.44	6.397	6.367	3.08	3.05	1.47	1.47
.3076	33.34	33.28	22.16	22.09	16.64	16.56	6.414	6.386	3.10	3.07	1.48	1.48
.3096	33.58	33.52	22.32	22.25	16.76	16.68	6.462	6.434	3.12	3.09	1.49	1.49
.3116	33.82	33.76	22.48	22.41	16.88	16.80	6.510	6.482	3.15	3.12	1.51	1.50
.3136	34.00	34.00	22.64	22.57	17.00	16.92	6.558	6.531	3.17	3.14	1.52	1.51
.3156	34.30	34.24	22.81	22.73	17.12	17.04	6.608	6.580	3.19	3.16	1.53	1.52
.3176	34.54	34.48	22.97	22.89	17.24	17.16	6.654	6.629	3.21	3.18	1.54	1.53
.3196	34.78	34.72	23.13	23.05	17.36	17.28	6.703	6.678	3.24	3.21	1.55	1.54
.3216	35.02	34.96	23.29	23.21	17.48	17.40	6.751	6.727	3.26	3.23	1.56	1.55
.3236	35.26	35.20	23.45	23.38	17.60	17.52	6.799	6.776	3.28	3.26	1.57	1.56
.3256	35.50	35.44	23.62	23.54	17.72	17.64	6.848	6.825	3.30	3.27	1.58	1.57
.3275	35.75	35.68	23.77	23.70	17.84	17.76	6.897	6.875	3.33	3.30	1.60	1.59
.3295	36.00	35.92	23.93	23.86	17.96	17.88	6.945	6.924	3.35	3.32	1.61	1.60
.3315	36.24	36.16	24.09	24.02	18.08	18.00	6.993	6.973	3.37	3.34	1.62	1.61
.3335	36.48	36.40	24.25	24.18	18.20	18.12	7.042	7.023	3.39	3.36	1.63	1.62
.3355	36.73	36.65	24.42	24.35	18.33	18.25	7.091	7.073	3.42	3.39	1.64	1.63
.3375	36.97	36.89	24.58	24.51	18.45	18.37	7.139	7.122	3.44	3.41	1.65	1.64
.3395	37.22	37.13	24.74	24.67	18.57	18.49	7.188	7.172	3.46	3.43	1.66	1.65
.3415	37.47	37.37	24.90	24.83	18.69	18.61	7.237	7.222	3.48	3.45	1.67	1.66
.3435	37.72	37.62	25.07	25.00	18.82	18.74	7.286	7.272	3.51	3.48	1.69	1.68
.3455	37.96	37.86	25.23	25.16	18.94	18.86	7.334	7.321	3.53	3.50	1.70	1.69
.3475	38.21	38.10	25.40	25.33	19.06	18.99	7.383	7.371	3.55	3.52	1.71	1.70
.3495	38.46	38.44	25.56	25.49	19.19	19.12	7.432	7.421	3.58	3.55	1.72	1.71

TABLE 41  
EXPANDED MEISSEL-HILLER TABLE FOR INVERT SUGAR IN CANE JUICE

Grams Juice in in 100 ML.		10			20			30			40		
Polarization		10	14	18	10	14	18	10	14	18	10	14	18
		Percentages of Glucose											
Cu Gram	CuO Gram	1.55	1.55	1.52	0.75	0.73	0.72	0.49	0.46	0.46	0.36	0.35	0.33
.150	.1877	1.81	1.60	1.58	0.78	0.76	0.75	0.51	0.50	0.49	0.37	0.36	0.34
.155	.1939	1.66	1.65	1.63	0.81	0.78	0.77	0.52	0.51	0.50	0.37	0.36	0.35
.160	.2003	1.71	1.70	1.69	0.83	0.82	0.80	0.54	0.53	0.52	0.39	0.38	0.36
.165	.2065	1.76	1.76	1.75	0.86	0.85	0.84	0.56	0.55	0.53	0.41	0.40	0.38
.170	.2128	1.82	1.81	1.80	0.89	0.87	0.84	0.58	0.57	0.55	0.43	0.42	0.39
.175	.2191	1.87	1.87	1.86	0.92	0.90	0.87	0.60	0.58	0.57	0.44	0.43	0.40
.180	.2252	1.93	1.92	1.91	0.94	0.93	0.90	0.63	0.60	0.59	0.45	0.44	0.41
.185	.2315	1.98	1.98	1.98	0.97	0.95	0.93	0.64	0.62	0.60	0.47	0.46	0.43
.190	.2378	2.03	2.03	2.03	1.00	0.98	0.96	0.65	0.64	0.62	0.48	0.46	0.43
.195	.2440	2.08	2.08	2.08	1.03	1.01	0.99	0.67	0.66	0.65	0.49	0.48	0.45
.200	.2503	...	...	...	1.06	1.04	1.03	0.69	0.68	0.67	0.51	0.50	0.49
.205	.2566	...	...	...	1.09	1.07	1.06	0.71	0.69	0.68	0.52	0.50	0.49
.210	.2628	...	...	...	1.12	1.10	1.09	0.73	0.71	0.70	0.54	0.53	0.51
.215	.2690	...	...	...	1.15	1.12	1.11	0.76	0.72	0.72	0.55	0.54	0.53
.220	.2754	...	...	...	1.18	1.14	1.13	0.78	0.74	0.74	0.56	0.55	0.54
.225	.2817	...	...	...	1.21	1.18	1.16	0.80	0.77	0.76	0.57	0.57	0.56
.230	.2879	...	...	...	1.24	1.21	1.19	0.82	0.79	0.78	0.59	0.59	0.57
.235	.2942	...	...	...	1.26	1.24	1.22	0.84	0.82	0.80	0.61	0.60	0.58
.240	.3004	...	...	...	1.29	1.27	1.25	0.86	0.84	0.82	0.62	0.61	0.60
.245	.3066	...	...	...	1.32	1.30	1.28	0.88	0.85	0.83	0.63	0.62	0.61
.250	.3129	...	...	...	1.35	1.32	1.31	0.90	0.87	0.85	0.65	0.64	0.63
.255	.3192	...	...	...	1.37	1.34	1.34	0.92	0.89	0.88	0.66	0.65	0.63
.260	.3254	...	...	...	1.40	1.37	1.36	0.93	0.90	0.89	0.67	0.67	0.65
.265	.3317	...	...	...	1.43	1.41	1.39	0.95	0.92	0.91	0.69	0.68	0.66
.270	.3379	...	...	...	1.46	1.44	1.42	0.97	0.94	0.93	0.71	0.69	0.68
.275	.3441	...	...	...	1.48	1.47	1.45	0.99	0.96	0.96	0.72	0.70	0.71
.280	.3504	...	...	...	1.51	1.50	1.48	1.01	0.98	0.96	0.74	0.72	0.71
.285	.3568	...	...	...	1.54	1.52	1.51	1.03	0.99	0.99	0.76	0.74	0.73
.290	.3630	...	...	...	1.57	1.55	1.54	1.05	1.01	1.01	0.77	0.75	0.75
.295	.3692	...	...	...	1.59	1.58	1.56	1.05	1.03	1.02	0.78	0.77	0.76
.300	.3755	...	...	...	1.59	1.58	1.56	1.05	1.03	1.02	0.78	0.77	0.76

TABLE 42  
EXPANDED MEISSL-HILLER TABLE FOR INVERT SUGAR IN CANE SIRUP

Grams Sirup in 100 Ml.		4				6				10			
		45.0	50.0	55.0	60.0	45.0	50.0	55.0	60.0	45.0	50.0	55.0	60.0
Polarization		Percentages of Glucose											
Cu Gram	CuO Gram												
.150	.1877	3.79	3.77	3.75	3.73	2.46	2.43	2.41	2.39	1.40	1.38	1.36	1.34
.155	.1939	3.09	3.01	2.89	3.87	2.56	2.53	2.51	2.49	1.46	1.44	1.42	1.40
.160	.2003	4.07	4.04	4.02	4.00	2.64	2.62	2.59	2.57	1.51	1.49	1.48	1.45
.165	.2065	4.21	4.18	4.16	4.14	2.74	2.72	2.69	2.67	1.57	1.55	1.54	1.51
.170	.2128	4.35	4.32	4.30	4.27	2.83	2.80	2.78	2.76	1.62	1.61	1.59	1.57
.175	.2191	4.49	4.46	4.44	4.41	2.93	2.90	2.88	2.86	1.68	1.67	1.65	1.63
.180	.2252	4.63	4.60	4.57	4.55	3.01	2.99	2.96	2.94	1.74	1.72	1.70	1.69
.185	.2315	4.77	4.74	4.71	4.69	3.11	3.09	3.06	3.04	1.80	1.78	1.76	1.75
.190	.2378	4.92	4.88	4.85	4.83	3.20	3.18	3.15	3.12	1.85	1.83	1.82	1.80
.195	.2440	5.06	5.02	4.99	4.97	3.30	3.28	3.25	3.22	1.91	1.89	1.88	1.86
.200	.2503	5.21	5.17	5.13	5.11	3.38	3.36	3.34	3.31	1.96	1.95	1.93	1.92
.205	.2566	5.35	5.31	5.27	5.25	3.48	3.46	3.44	3.41	2.02	2.01	2.00	1.98
.210	.2628	5.48	5.46	5.42	5.39	3.56	3.54	3.53	3.50	2.07	2.06	2.05	2.03
.215	.2690	5.62	5.60	5.56	5.53	3.66	3.64	3.63	3.60	2.13	2.12	2.11	2.09
.220	.2754	5.75	5.75	5.70	5.67	3.75	3.73	3.71	3.69	2.18	2.17	2.16	2.15
.225	.2817	5.89	5.89	5.84	5.81	3.85	3.83	3.81	3.79	2.24	2.23	2.22	2.21
.230	.2879	6.03	6.03	5.99	5.96	3.94	3.91	3.89	3.88	2.29	2.28	2.27	2.26
.235	.2942	6.17	6.17	6.13	6.10	4.04	4.01	3.99	3.98	2.35	2.34	2.33	2.32
.240	.3004	6.30	6.30	6.29	6.26	4.13	4.10	4.08	4.06	2.41	2.40	2.39	2.38
.245	.3066	6.44	6.44	6.43	6.39	4.23	4.20	4.18	4.16	2.47	2.46	2.45	2.44
.250	.3129	6.58	6.58	6.58	6.54	4.34	4.29	4.27	4.25	2.52	2.51	2.50	2.49
.255	.3192	6.72	6.72	6.72	6.68	4.44	4.39	4.37	4.35	2.57	2.57	2.56	2.55
.260	.3254	6.86	6.85	6.85	6.84	4.52	4.48	4.46	4.44	2.63	2.62	2.61	2.60
.265	.3317	7.00	6.99	6.99	6.98	4.62	4.58	4.56	4.54	2.69	2.68	2.67	2.66
.270	.3379	7.13	7.13	7.13	7.13	4.71	4.67	4.65	4.64	2.75	2.74	2.73	2.72
.275	.3441	7.27	7.27	7.27	7.27	4.81	4.77	4.75	4.74	2.81	2.80	2.79	2.78
.280	.3504	7.41	7.41	7.41	7.41	4.91	4.87	4.84	4.83	2.87	2.85	2.84	2.83
.285	.3568	7.55	7.55	7.55	7.55	5.01	4.97	4.94	4.93	2.93	2.91	2.90	2.89
.290	.3630	7.70	7.69	7.69	7.69	5.11	5.07	5.03	5.02	2.98	2.97	2.96	2.95
.295	.3692	7.84	7.83	7.83	7.83	5.21	5.17	5.13	5.12	3.04	3.03	3.02	3.01
.300	.3755	7.98	7.97	7.97	7.97	5.31	5.27	5.23	5.21	3.10	3.09	3.08	3.07

TABLE 43

HERZFELD'S TABLE FOR DETERMINING INVERT SUGAR IN RAW SUGARS  
(INVERT SUGAR NOT TO EXCEED 1.5 PER CENT)

Copper (Cu), Mgs.	Invert Sugar, Per Cent	Copper (Cu), Mgs.	Invert Sugar, Per Cent	Copper (Cu), Mgs.	Invert Sugar, Per Cent	Copper (Cu), Mgs.	Invert Sugar, Per Cent	Copper (Cu), Mgs.	Invert Sugar, Per Cent
50	0.050	105	0.325	160	0.621	215	0.929	270	1.242
51	0.054	106	0.330	161	0.627	216	0.935	271	1.248
52	0.058	107	0.335	162	0.633	217	0.940	272	1.253
53	0.062	108	0.340	163	0.639	218	0.946	273	1.259
54	0.066	109	0.346	164	0.645	219	0.951	274	1.265
55	0.070	110	0.351	165	0.651	220	0.957	275	1.271
56	0.074	111	0.356	166	0.657	221	0.962	276	1.276
57	0.078	112	0.361	167	0.663	222	0.968	277	1.282
58	0.082	113	0.366	168	0.669	223	0.973	278	1.288
59	0.086	114	0.371	169	0.675	224	0.979	279	1.294
60	0.090	115	0.376	170	0.680	225	0.984	280	1.299
61	0.094	116	0.381	171	0.686	226	0.990	281	1.305
62	0.098	117	0.386	172	0.692	227	0.996	282	1.311
63	0.103	118	0.392	173	0.698	228	1.001	283	1.317
64	0.108	119	0.397	174	0.704	229	1.007	284	1.322
65	0.113	120	0.402	175	0.709	230	1.013	285	1.328
66	0.118	121	0.407	176	0.715	231	1.018	286	1.334
67	0.123	122	0.412	177	0.720	232	1.024	287	1.339
68	0.128	123	0.417	178	0.726	233	1.030	288	1.345
69	0.133	124	0.423	179	0.731	234	1.036	289	1.351
70	0.138	125	0.428	180	0.737	235	1.041	290	1.357
71	0.143	126	0.433	181	0.742	236	1.047	291	1.362
72	0.148	127	0.438	182	0.748	237	1.053	292	1.368
73	0.152	128	0.443	183	0.753	238	1.058	293	1.374
74	0.157	129	0.448	184	0.759	239	1.064	294	1.380
75	0.162	130	0.453	185	0.764	240	1.070	295	1.385
76	0.167	131	0.458	186	0.770	241	1.076	296	1.391
77	0.172	132	0.463	187	0.775	242	1.081	297	1.397
78	0.177	133	0.468	188	0.781	243	1.087	298	1.403
79	0.182	134	0.473	189	0.786	244	1.093	299	1.408
80	0.187	135	0.478	190	0.792	245	1.099	300	1.414
81	0.192	136	0.483	191	0.797	246	1.104	301	1.420
82	0.197	137	0.488	192	0.803	247	1.110	302	1.425
83	0.202	138	0.493	193	0.808	248	1.116	303	1.431
84	0.208	139	0.498	194	0.814	249	1.122	304	1.437
85	0.213	140	0.503	195	0.819	250	1.127	305	1.443
86	0.219	141	0.509	196	0.825	251	1.133	306	1.448
87	0.225	142	0.515	197	0.830	252	1.139	307	1.454
88	0.231	143	0.521	198	0.836	253	1.144	308	1.460
89	0.236	144	0.527	199	0.841	254	1.150	309	1.466
90	0.242	145	0.533	200	0.847	255	1.156	310	1.471
91	0.248	146	0.538	201	0.852	256	1.162	311	1.477
92	0.254	147	0.544	202	0.858	257	1.167	312	1.483
93	0.260	148	0.550	203	0.863	258	1.173	313	1.489
94	0.265	149	0.556	204	0.869	259	1.179	314	1.494
95	0.271	150	0.562	205	0.874	260	1.185	315	1.500
96	0.277	151	0.568	206	0.880	261	1.190		
97	0.283	152	0.574	207	0.885	262	1.196		
98	0.288	153	0.580	208	0.891	263	1.202		
99	0.294	154	0.586	209	0.896	264	1.207		
100	0.300	155	0.592	210	0.902	265	1.213		
101	0.305	156	0.598	211	0.907	266	1.219		
102	0.310	157	0.604	212	0.913	267	1.225		
103	0.315	158	0.609	213	0.918	268	1.231		
104	0.320	159	0.615	214	0.924	269	1.236		

TABLE 44

CONDENSED MUNSON AND WALKER'S TABLE FOR INVERT SUGAR ALONE AND INVERT SUGAR IN THE PRESENCE OF SUCROSE

(Expressed in Milligrams)

Cu- prous Oxide (Cu <sub>2</sub> O)	Cop- per (Cu)	In- vert Sugar	Invert Sugar and Sucrose		Cu- prous Oxide (Cu <sub>2</sub> O)	Cop- per (Cu)	In- vert Sugar	Invert Sugar and Sucrose	
			0.4 Gram Total Sugar	2 Grams Total Sugar				0.4 Gram Total Sugar	2 Grams Total Sugar
10	8.9	4.5	1.6	.....	250	222.1	116.4	115.1	109.0
15	13.3	6.7	3.9	.....	255	226.5	118.9	117.6	111.5
20	17.8	8.9	6.1	.....	260	231.0	121.4	120.1	114.0
25	22.2	11.2	8.4	.....	265	235.4	123.9	122.6	116.5
30	26.6	13.4	10.7	4.3	270	239.8	126.4	125.1	119.0
35	31.1	15.6	12.9	6.5	275	244.3	128.9	127.7	121.6
40	35.5	17.8	15.2	8.8	280	248.7	131.4	130.2	124.1
45	40.0	20.1	17.5	11.1	285	253.2	133.9	132.7	126.6
50	44.4	22.3	19.7	13.4	290	257.6	136.4	135.3	129.2
55	48.9	24.6	22.0	15.7	295	262.0	138.9	137.8	131.7
60	53.3	26.8	24.3	18.0	300	266.5	141.5	140.4	134.2
65	57.7	29.1	26.6	20.3	305	270.9	144.0	142.9	136.8
70	62.2	31.3	28.9	22.6	310	275.4	146.6	145.5	139.4
75	66.6	33.6	31.2	24.9	315	279.8	149.1	148.1	141.9
80	71.1	35.9	33.5	27.3	320	284.2	151.7	150.7	144.5
85	75.5	38.2	35.8	29.6	325	288.7	154.3	153.2	147.1
90	79.9	40.4	38.2	31.9	330	293.1	156.8	155.8	149.7
95	84.4	42.7	40.5	34.2	335	297.6	159.4	158.4	152.3
100	88.8	45.0	42.8	36.6	340	302.0	162.0	161.0	154.8
105	93.3	47.3	45.2	38.9	345	306.5	164.6	163.7	157.5
110	97.7	49.6	47.5	41.3	350	310.9	167.2	166.3	160.1
115	102.2	51.9	49.8	43.6	355	315.3	169.8	168.9	162.7
120	106.6	54.3	52.2	46.0	360	319.8	172.5	171.5	165.3
125	111.0	56.6	54.5	48.3	365	324.2	175.1	174.2	167.9
130	115.5	58.9	56.9	50.7	370	328.7	177.7	176.8	170.6
135	119.9	61.2	59.3	53.1	375	333.1	180.4	179.5	173.2
140	124.4	63.6	61.6	55.5	380	337.5	183.0	182.1	175.9
145	128.8	65.9	64.0	57.8	385	342.0	185.7	184.8	178.5
150	133.2	68.3	66.4	60.2	390	346.4	188.4	187.5	181.2
155	137.7	70.6	68.8	62.6	395	350.9	191.0	190.2	183.9
160	142.1	73.0	71.2	65.0	400	355.3	193.7	192.9	186.5
165	146.6	75.3	73.6	67.4	405	359.7	196.4	195.6	189.2
170	151.0	77.7	76.0	69.8	410	364.2	199.1	198.3	191.9
175	155.5	80.1	78.4	72.2	415	368.6	201.8	201.0	194.6
180	159.9	82.5	80.8	74.6	420	373.1	204.6	203.7	197.3
185	164.3	84.9	83.2	77.1	425	377.5	207.3	206.5	200.0
190	168.8	87.2	85.6	79.5	430	382.0	210.0	209.2	202.7
195	173.2	89.6	88.0	81.9	435	386.4	212.8	212.0	205.5
200	177.7	92.0	90.5	84.4	440	390.8	215.5	214.7	208.2
205	182.1	94.5	92.9	86.8	445	395.3	218.3	217.5	211.0
210	186.5	96.9	95.4	89.2	450	399.7	221.1	220.2	213.7
215	191.0	99.3	97.8	91.7	455	404.2	223.9	223.0	216.5
220	195.4	101.7	100.3	94.2	460	408.6	226.7	225.8	219.2
225	199.9	104.2	102.7	96.6	465	413.0	229.5	228.6	222.0
230	204.3	106.6	105.2	99.1	470	417.5	232.3	231.4	224.8
235	208.7	109.1	107.7	101.6	475	421.9	235.1	234.2	227.6
240	213.2	111.5	110.1	104.0	480	426.4	237.9	237.1	230.3
245	217.6	114.0	112.6	106.5	485	430.8	240.8	239.9	233.2
...	.....	.....	.....	.....	490	435.3	243.6	242.7	236.0

TABLE 45  
INVERT SUGAR TABLE FOR 10 ML. OF FEHLING'S SOLUTION—EYNON AND LANE METHOD  
SOLUTIONS CONTAINING, BESIDES INVERT SUGAR

Ml. of Sugar Solution Required	No Sucrose		1 Gm. Sucrose per 100 ML.		5 Gm. Sucrose per 100 ML.		10 Gm. Sucrose per 100 ML.		25 Gm. Sucrose per 100 ML.	
	Invert Sugar Factor *	Mg. Invert Sugar per 100 ML.	Invert Sugar Factor *	Mg. Invert Sugar per 100 ML.	Invert Sugar Factor *	Mg. Invert Sugar per 100 ML.	Invert Sugar Factor *	Mg. Invert Sugar per 100 ML.	Invert Sugar Factor *	Mg. Invert Sugar per 100 ML.
15	50.5	336	49.9	333	47.6	317	46.1	307	43.4	289
16	50.6	316	50.0	312	47.6	297	46.1	288	43.4	271
17	50.7	298	50.1	295	47.6	280	46.1	271	43.4	255
18	50.8	282	50.1	278	47.6	264	46.1	256	43.3	240
19	50.8	267	50.2	264	47.6	250	46.1	243	43.3	227
20	50.9	254.5	50.2	251.0	47.6	238.0	46.1	230.5	43.2	216
21	51.0	242.9	50.2	239.0	47.6	228.7	46.1	219.5	43.2	208
22	51.0	231.8	50.3	228.2	47.6	216.4	46.1	209.5	43.1	196
23	51.1	222.2	50.3	218.7	47.6	207.0	46.1	200.4	43.0	187
24	51.2	213.3	50.3	209.8	47.6	198.3	46.1	192.1	42.9	179
25	51.2	204.8	50.4	201.6	47.6	190.4	46.0	184.0	42.8	171
26	51.3	197.4	50.4	193.8	47.6	183.1	46.0	176.9	42.8	164
27	51.4	190.4	50.4	186.7	47.6	176.4	46.0	170.4	42.7	158
28	51.4	183.7	50.5	180.2	47.7	170.3	46.0	164.3	42.7	152
29	51.5	177.6	50.5	174.1	47.7	164.5	46.0	158.6	42.6	147
30	51.5	171.7	50.5	168.3	47.7	159.0	46.0	153.3	42.5	142
31	51.6	166.3	50.6	163.1	47.7	153.9	45.9	148.1	42.5	137
32	51.6	161.2	50.6	158.1	47.7	149.1	45.9	143.4	42.4	132
33	51.7	156.6	50.6	153.3	47.7	144.5	45.9	139.1	42.3	128
34	51.7	152.2	50.6	148.9	47.7	140.3	45.8	134.9	42.2	124
35	51.8	147.0	50.7	144.7	47.7	136.3	45.8	130.9	42.2	121
36	51.8	143.9	50.7	140.7	47.7	132.5	45.8	127.1	42.1	117
37	51.9	140.2	50.7	137.0	47.7	128.9	45.7	123.5	42.0	114
38	51.9	136.6	50.7	133.5	47.7	125.5	45.7	120.3	42.0	111
39	52.0	133.3	50.8	130.2	47.7	122.3	45.7	117.1	41.9	107
40	52.0	130.1	50.8	127.0	47.7	119.2	45.6	114.1	41.8	104
41	52.1	127.1	50.8	123.9	47.7	116.3	45.6	111.2	41.8	102
42	52.1	124.2	50.8	121.0	47.7	113.5	45.6	108.5	41.7	99
43	52.2	121.4	50.8	118.2	47.7	110.9	45.5	105.8	41.6	97
44	52.2	118.7	50.9	115.6	47.7	108.4	45.5	103.4	41.5	94

\* Mg. of invert sugar corresponding to 10 ml. of Fehling's solution.

TABLE 46

TABLE 46

INVERT SUGAR TABLE FOR 25 ML. OF FEHLING'S SOLUTION

SOLUTIONS CONTAINING, BESIDES INVERT SUGAR

Ml. of Sugar Solution Required	No Sucrose		1 Gm. Sucrose per 100 ML.	
	Invert Sugar Factor *	Mg. Invert Sugar per 100 ML.	1 Gm. Invert Sugar Factor *	Mg. Invert Sugar per 100 ML.
15	123.6	824	122.6	817
16	123.6	772	122.7	767
17	123.6	727	122.7	721
18	123.7	687	122.7	682
19	123.7	651	122.8	646
20	123.8	619.0	122.8	614.0
21	123.8	589.5	122.8	584.8
22	123.9	563.2	122.9	558.2
23	123.9	538.7	122.9	534.0
24	124.0	516.7	122.9	512.1
25	124.0	496.0	123.0	492.0
26	124.1	477.3	123.0	473.1
27	124.1	459.7	123.0	455.6
28	124.2	443.6	123.1	439.6
29	124.2	428.3	123.1	424.4
30	124.3	414.3	123.1	410.4
31	124.3	401.0	123.2	397.4
32	124.4	388.7	123.2	385.0
33	124.4	377.0	123.2	373.4
34	124.5	366.2	123.3	362.6
35	124.5	355.8	123.3	352.3
36	124.6	346.1	123.3	342.5
37	124.6	336.8	123.4	333.5
38	124.7	328.1	123.4	324.7
39	124.7	319.7	123.4	316.4
40	124.8	311.9	123.4	308.6
41	124.8	304.4	123.5	301.2
42	124.9	297.3	123.5	294.1
43	124.9	290.5	123.5	287.3
44	125.0	284.1	123.6	280.9
45	125.0	277.9	123.6	274.7
46	125.1	272.0	123.6	268.7
47	125.1	266.3	123.7	263.1
48	125.2	260.8	123.7	257.7
49	125.2	255.5	123.7	252.5
50	125.3	250.6	123.8	247.6

\*Mg. of invert sugar corresponding to 25 ml. of Fehling's solution.



TABLE 47

RECIPROCAL OF NUMBERS FROM 11 TO 36, ADVANCING BY TENTHS \*

Num- ber	Recip- rocal	Num- ber	Recip- rocal	Num- ber	Recip- rocal	Num- ber	Recip- rocal	Num- ber	Recip- rocal
<b>11.0</b>	.0909	<b>16.0</b>	.0625	<b>21.0</b>	.0476	<b>26.0</b>	.0385	<b>31.0</b>	.0322
.1	.0900	.1	.0621	.1	.0474	.1	.0383	.1	.0321
.2	.0893	.2	.0617	.2	.0472	.2	.0381	.2	.0320
.3	.0885	.3	.0613	.3	.0469	.3	.0380	.3	.0319
.4	.0877	.4	.0610	.4	.0467	.4	.0379	.4	.0318
.5	.0869	.5	.0606	.5	.0465	.5	.0377	.5	.0317
.6	.0862	.6	.0602	.6	.0463	.6	.0376	.6	.0316
.7	.0855	.7	.0599	.7	.0461	.7	.0374	.7	.0315
.8	.0847	.8	.0595	.8	.0459	.8	.0373	.8	.0314
.9	.0840	.9	.0592	.9	.0457	.9	.0372	.9	.0313
<b>12.0</b>	.0833	<b>17.0</b>	.0588	<b>22.0</b>	.0454	<b>27.0</b>	.0370	<b>32.0</b>	.0312
.1	.0826	.1	.0585	.1	.0452	.1	.0369	.1	.0311
.2	.0820	.2	.0581	.2	.0450	.2	.0368	.2	.0310
.3	.0813	.3	.0578	.3	.0448	.3	.0366	.3	.0309
.4	.0806	.4	.0575	.4	.0446	.4	.0365	.4	.0308
.5	.0800	.5	.0571	.5	.0444	.5	.0364	.5	.0308
.6	.0794	.6	.0568	.6	.0442	.6	.0362	.6	.0307
.7	.0787	.7	.0565	.7	.0440	.7	.0361	.7	.0305
.8	.0781	.8	.0562	.8	.0438	.8	.0360	.8	.0305
.9	.0775	.9	.0559	.9	.0437	.9	.0358	.9	.0304
<b>13.0</b>	.0769	<b>18.0</b>	.0555	<b>23.0</b>	.0435	<b>28.0</b>	.0357	<b>33.0</b>	.0303
.1	.0763	.1	.0552	.1	.0432	.1	.0356	.1	.0302
.2	.0757	.2	.0549	.2	.0431	.2	.0355	.2	.0301
.3	.0752	.3	.0546	.3	.0429	.3	.0353	.3	.0300
.4	.0746	.4	.0543	.4	.0427	.4	.0352	.4	.0299
.5	.0741	.5	.0540	.5	.0425	.5	.0351	.5	.0298
.6	.0735	.6	.0538	.6	.0424	.6	.0350	.6	.0297
.7	.0730	.7	.0535	.7	.0422	.7	.0348	.7	.0296
.8	.0725	.8	.0532	.8	.0420	.8	.0347	.8	.0295
.9	.0719	.9	.0529	.9	.0418	.9	.0346	.9	.0295
<b>14.0</b>	.0714	<b>19.0</b>	.0526	<b>24.0</b>	.0417	<b>29.0</b>	.0345	<b>34.0</b>	.0294
.1	.0709	.1	.0523	.1	.0415	.1	.0344	.1	.0293
.2	.0704	.2	.0521	.2	.0413	.2	.0342	.2	.0292
.3	.0699	.3	.0518	.3	.0411	.3	.0341	.3	.0291
.4	.0694	.4	.0515	.4	.0409	.4	.0340	.4	.0290
.5	.0690	.5	.0513	.5	.0408	.5	.0339	.5	.0289
.6	.0685	.6	.0510	.6	.0406	.6	.0338	.6	.0289
.7	.0680	.7	.0508	.7	.0405	.7	.0337	.7	.0288
.8	.0676	.8	.0505	.8	.0403	.8	.0335	.8	.0287
.9	.0671	.9	.0502	.9	.0402	.9	.0334	.9	.0286
<b>15.0</b>	.0667	<b>20.0</b>	.0500	<b>25.0</b>	.0400	<b>30.0</b>	.0333	<b>35.0</b>	.0285
.1	.0662	.1	.0497	.1	.0398	.1	.0332	.1	.0284
.2	.0658	.2	.0495	.2	.0397	.2	.0331	.2	.0284
.3	.0654	.3	.0493	.3	.0395	.3	.0330	.3	.0283
.4	.0649	.4	.0490	.4	.0394	.4	.0329	.4	.0282
.5	.0645	.5	.0488	.5	.0392	.5	.0328	.5	.0282
.6	.0641	.6	.0485	.6	.0391	.6	.0327	.6	.0281
.7	.0637	.7	.0483	.7	.0389	.7	.0326	.7	.0280
.8	.0633	.8	.0481	.8	.0388	.8	.0325	.8	.0279
.9	.0629	.9	.0478	.9	.0386	.9	.0324	.9	.0278

\* See page 250 for suggestions relative to the use of this table.

TABLE 48

## JACKSON-GILLIS CLERGET METHOD IV

(See page 234)

(Bureau of Standards Sci. Papers No. 375)

Column No. 1 is the algebraic sum of  $P - P'$  corrected to normality. Column No. 2 is the Clerget Divisor corresponding to that value of  $P - P'$ . Apply temperature correction for exact temperature at which the invert polarization was read. Corrected Clerget Divisor divided into  $100(P - P') = \text{Clerget Sucrose}$ .

## CLERGET DIVISOR

No. 1	No. 2	No. 1	No. 2	No. 1	No. 2	No. 1	No. 2
5	131.78	40	132.01	77	132.26	117	132.52
7	131.80	45	132.04	80	132.28	120	132.54
10	131.82	50	132.08	83	132.29	122	132.56
12	131.83	55	132.11	85	132.31	125	132.58
15	131.85	60	132.15	87	132.33	127	132.59
17	131.87	63	132.16	90	132.35	130	132.61
20	131.89	65	132.18	95	132.38	132.63	132.63
22	131.90	67	132.19	100	132.41	133	132.63
25	131.92	70	132.21	105	132.44		
30	131.95	72	132.22	110	132.47		
35	131.98	75	132.24	115	132.51		

## SUBTRACTION TEMPERATURE CORRECTIONS

C°	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
20	0.00	0.05	0.11	0.16	0.21	0.27	0.32	0.37	0.42	0.48
21	0.53	0.58	0.64	0.69	0.74	0.80	0.85	0.90	0.95	1.01
22	1.06	1.11	1.17	1.22	1.27	1.33	1.38	1.43	1.48	1.54
23	1.59	1.64	1.70	1.75	1.80	1.86	1.91	1.96	2.01	2.07
24	2.12	2.17	2.23	2.28	2.33	2.39	2.44	2.49	2.54	2.60
25	2.65	2.70	2.76	2.81	2.86	2.92	2.97	3.02	3.07	3.13
26	3.18	3.23	3.29	3.34	3.39	3.44	3.50	3.55	3.60	3.66
27	3.71	3.76	3.82	3.87	3.92	3.98	4.03	4.08	4.13	4.19
28	4.24	4.29	4.35	4.40	4.45	4.51	4.56	4.61	4.66	4.72
29	4.77	4.82	4.88	4.93	4.98	5.04	5.09	5.14	5.19	5.25
30	5.30	5.35	5.41	5.46	5.51	5.57	5.62	5.67	5.72	5.78
31	5.83	5.88	5.94	5.99	6.04	6.10	6.15	6.20	6.25	6.31
32	6.36	6.41	6.47	6.52	6.57	6.63	6.68	6.73	6.78	6.84
33	6.89	6.94	7.00	7.05	7.10	7.16	7.21	7.26	7.31	7.37
34	7.42	7.47	7.53	7.58	7.63	7.69	7.74	7.79	7.84	7.90
35	7.95	8.00	8.06	8.11	8.16	8.22	8.27	8.32	8.37	8.43

TABLE 49  
CALCULATING THE PER CENT SUCROSE IN BAGASSE—(SPENCER)

Conditions: 100 gm. of bagasse, 1000 gm. water; dry lead clarification; 400 mm. observation tube;  $W = (100 + \text{Water}) - \% \text{ fiber (marc)}$

W (Grams)	980	990	1000	1010	1020	1030	1040	1050	1060	1070	1080
Observation	Per Cent Sucrose (Polarization) in the Bagasse										
1.5	1.91	1.93	1.95	1.97	1.99	2.01	2.03	2.05	2.07	2.09	2.11
.6	2.04	2.06	2.08	2.10	2.12	2.14	2.16	2.18	2.20	2.23	2.25
.7	2.17	2.19	2.21	2.23	2.25	2.28	2.30	2.32	2.34	2.36	2.39
.8	2.29	2.32	2.34	2.36	2.39	2.41	2.43	2.45	2.48	2.50	2.53
.9	2.42	2.45	2.47	2.49	2.52	2.54	2.57	2.59	2.62	2.64	2.67
2.0	2.55	2.57	2.60	2.63	2.65	2.68	2.70	2.73	2.76	2.78	2.81
.1	2.68	2.70	2.73	2.76	2.78	2.81	2.84	2.87	2.89	2.92	2.95
.2	2.80	2.83	2.86	2.89	2.92	2.95	2.97	3.00	3.03	3.06	3.09
.3	2.93	2.96	2.99	3.02	3.05	3.08	3.11	3.14	3.17	3.20	3.23
.4	3.06	3.09	3.12	3.15	3.18	3.21	3.24	3.28	3.31	3.34	3.37
.5	3.19	3.22	3.25	3.28	3.32	3.35	3.38	3.41	3.45	3.48	3.51
.6	3.31	3.35	3.38	3.41	3.45	3.48	3.52	3.55	3.58	3.62	3.65
.7	3.44	3.47	3.51	3.55	3.58	3.62	3.65	3.69	3.73	3.76	3.79
.8	3.57	3.60	3.64	3.68	3.71	3.75	3.79	3.82	3.86	3.89	3.93
.9	3.69	3.73	3.77	3.81	3.85	3.88	3.92	3.96	4.00	4.03	4.07
3.0	3.82	3.86	3.90	3.94	3.98	4.02	4.06	4.10	4.13	4.17	4.21
.1	3.95	3.99	4.03	4.07	4.11	4.15	4.19	4.23	4.27	4.31	4.35
.2	4.08	4.12	4.16	4.20	4.24	4.28	4.33	4.37	4.41	4.45	4.49
.3	4.20	4.25	4.29	4.33	4.38	4.42	4.46	4.50	4.55	4.59	4.63
.4	4.33	4.38	4.42	4.46	4.51	4.55	4.60	4.64	4.69	4.73	4.77
.5	4.46	4.50	4.55	4.60	4.64	4.68	4.73	4.78	4.82	4.87	4.91
.6	4.59	4.63	4.68	4.73	4.77	4.82	4.87	4.91	4.96	5.01	5.05
.7	4.71	4.76	4.81	4.86	4.91	4.95	5.00	5.05	5.10	5.15	5.19
.8	4.84	4.89	4.94	4.99	5.04	5.09	5.14	5.19	5.24	5.29	5.34
.9	4.97	5.02	5.07	5.12	5.17	5.22	5.27	5.32	5.37	5.42	5.48
4.0	5.10	5.15	5.20	5.25	5.30	5.35	5.41	5.45	5.51	5.56	5.62
.1	5.22	5.28	5.33	5.38	5.44	5.49	5.54	5.60	5.65	5.70	5.76
.2	5.35	5.41	5.46	5.51	5.57	5.62	5.68	5.73	5.79	5.84	5.90
.3	5.48	5.55	5.59	5.65	5.70	5.76	5.81	5.87	5.93	5.98	6.04
.4	5.61	5.66	5.72	5.78	5.83	5.89	5.95	6.01	6.06	6.12	6.18

TABLE 50—Section A

SHOWING THE MORE IMPORTANT CHEMICAL AND PHYSICAL PROPERTIES OF THE CARBOHYDRATES—(Compiled by ERVIN E. EWELL)  
(From von Lippmann's "Chemie der Zuckerarten," second edition; Tollens' "Handbuch der Kohlenhydrate," with reference to original papers.)

Name, Synonyms, and Formulae	Melting-point °C.	Melting-point of Osazone °C.	Source, etc.
I. MONOSACCHARIDES			
A. BROSSES			
Glycollic aldehyde.....		169°-170°	Obtained by oxidation of glycol with nitric acid or reduction of glyoxal with zinc and acetic acid. Has not been obtained in a pure state.
B. TETRACOSMS			
Glycerose.....	Sirupy	131°; 200°	Obtained by oxidation of glycerol with nitric acid or with bromine. A mixture of diosacetone and glyceraldehyde.
C. TETRACOSMS			
Erythrose.....		1166°-168°	Made from tetrahydic alcohol erythrol (erythrite) by oxidation with nitric acid. Probably mixed with isomeric ketone $\text{CH}_2\text{OH}\cdot\text{CHOH}\cdot\text{CO}\cdot\text{CH}_2\text{OH}$ . Only its osazone has been obtained pure. Erythrol occurs free and combined in lichens and algae.
D. PENTACOSMS			
1. <i>Auto-pentoses</i>			
$\text{C}_6\text{H}_{10}\text{O}_5 = \text{CH}_2\text{OH}\cdot(\text{CHOH})_3\cdot\text{CHO}$	160°; 150° 430°	180°	Obtained alone or mixed with various other sugars by hydrolysis of gums and other vegetable substances.
<i>l</i> -arabinose.....	160°	159°-160° 163°	Obtained by synthesis.
pectinose, pectin-sugar.....			Obtained by mixing equal weights of <i>l</i> -arabinose and <i>d</i> -arabinose.
<i>d</i> -arabinose.....	135°-140° 141°; 7144° 1514°; 1154° 150°-163°	1152°-155° 155°; 10158° 1260°; 2.161° 1570°	Obtained alone or mixed with various other sugars by the hydrolysis of gums and other vegetable substances. Its most important source is a wood gum (xylan), extracted from various kinds of wood by means of dilute solutions of caustic alkalies.
xylose.....			This name has been given by Fischer to a synthetic sugar of which he prepared the osazone.
wood-sugar.....			Obtained by the reduction of the lactone of ribonic acid. Not yet known with certainty, but their existence is evidenced by the work of Fischer.
<i>t</i> -xylose.....		210°-215°	
Inactive xylose.....	Sirup		
Ribose.....			
2. <i>Keto-pentoses</i>			
$\text{C}_6\text{H}_8\text{O}_5 = (\text{CHOH})_3\cdot\text{CO}\cdot\text{CH}_2\text{OH}$			
3. <i>Methyl-pentoses</i>			
$\text{C}_7\text{H}_{12}\text{O}_5 = \text{C}_6\text{H}_5\text{O}_5\cdot\text{CH}_3$			
Fucose.....		159° 180°	From the seaweed <i>Fucus nodosus</i>
Rhamnose.....			Widely distributed in the vegetable kingdom in the form of various glucosides.
Isodulcitol rhamnolulicite, rhamn-nigin-sugar, hesperidin-sugar.	1304°-105°		

Chinovose. $C_6H_{10}O_5 = CH_2 \cdot (CHOH)_4 \cdot CHO$ E. Hexoses	Slurp	193°-194°	A decomposition product of chinovite, a constituent of china bark.
1. <i>Aldo-hexoses</i>			
<i>d</i> -glucose..... Dextrose, grape-sugar, starch-sugar.....	14144°-146° 1580-90° 141°-143°	205°-210° 208°	Free and combined in the form of glucosides, gums, and polysaccharides, and in mixtures with various other sugars in a host of vegetable spines and in animal secretions.
<i>l</i> -glucose.....	Slurp	210°-217°	Reduction of the lactone of <i>l</i> -gluconic acid.
<i>l</i> -glucose.....	Slurp	19205°-210°	Obtained by mixing equal quantities of <i>d</i> - and <i>l</i> -glucoses.
Inactive glucose.....	Slurp	19208°	Oxidation of manite; hydrolysis of various vegetable materials.
<i>d</i> -mannose.....	Slurp	19210°-217°	Obtained synthetically by reduction of <i>l</i> -mannonic acid.
Mannose, dextrorotatory mannose, isomannitose, semimannose.....	Slurp	156° 157°	Obtained by the reduction of the lactone of <i>l</i> -mannonic acid.
<i>l</i> -mannose.....	Slurp	198°-197°	Obtained by the reduction of <i>d</i> -gulonic acid, which is in turn obtained by the reduction of glycucronic acid or <i>d</i> -saccharic acid.
Inactive mannose.....	Slurp	192°-195° 208°	Obtained synthetically by the reduction of <i>l</i> -gulonic acid.
<i>d</i> -glucose.....	Slurp	198°-197°	Obtained by the reduction of <i>d</i> -idonic acid, which is obtained by heating <i>d</i> -gulonic acid with pyridin. Yields same osazone as <i>d</i> -glucose.
Dextrorotatory glucose.....	Slurp	198°-197°	Obtained from <i>l</i> -glucose in same manner that <i>d</i> -gulonic acid yields <i>d</i> -idose.
<i>l</i> -glucose.....	Slurp	198°-197°	Existence of <i>l</i> -idose is reported by Fischer.
<i>l</i> -glucose.....	Slurp	198°-197°	Rarely reported free in nature, but is widely distributed in combination. It is obtained pure or mixed by the hydrolysis of milk sugar, various glucosides and polysaccharides.
<i>d</i> -idose.....	Slurp	198°-197°	Obtained synthetically.
<i>l</i> -idose.....	Slurp	198°-197°	Obtained synthetically.
<i>l</i> -idose.....	Slurp	198°-197°	Obtained synthetically.
<i>d</i> -galactose.....	Slurp	198°-197°	Obtained synthetically.
galactose, dextrorotatory galactose.....	Slurp	198°-197°	Obtained synthetically.
<i>l</i> -galactose.....	Slurp	198°-197°	Obtained synthetically.
<i>l</i> -galactose.....	Slurp	198°-197°	Obtained synthetically.
<i>d</i> -talose.....	Slurp	198°-197°	Obtained synthetically.
<i>l</i> -talose.....	Slurp	198°-197°	Obtained synthetically.
<i>l</i> -talose.....	Slurp	198°-197°	Obtained synthetically.
Chitose.....	Slurp	198°-197°	Obtained synthetically.

1 Fischer and Landsteiner. 2 Scheibler, Lippmann, Conrad, and Guthzeit. 3 Ost. 4 Martin. 5 Bauer. 6 Bertrand. 7 Wheeler and Tollens. 8 Allen and Tollens. 9 Tollens. 10 Stone and Test. 11 Hébert. 12 Koch. 13 Behrend and Wtl. 14 Anhydride. 15 Hydrate. 16 The *d*-, *l*- and *t*-mannoses form *d*-, *l*- and *t*-glucosazones, respectively.

TABLE 50—Section A—Continued  
SHOWING THE MORE IMPORTANT CHEMICAL AND PHYSICAL PROPERTIES OF THE CARBOHYDRATES—(COMPILED BY ERVIN E. EWELL)

Name, Synonyms, and Formulae	Melting-point °C.	Melting-point of Osazone °C.	Source, etc.
2. <i>Keto-hexoses</i> $\text{CH}_2\text{OH} \cdot (\text{CHOH})_4 \cdot \text{CO} \cdot \text{CH}_2\text{OH}$ <i>d</i> -fructose..... fructose, levulose, fruit-sugar, chylarlose <i>l</i> -fructose..... <i>l</i> -fructose..... Inactive fructose, <i>l</i> -levulose, <i>e</i> -acrose Sorbitolose Sorbitose, sorbine..... Formose.....	95°-105° ..... Sirup ..... ..... Sirup .....	..... ..... ..... ..... 164° 144° 204°-206° .....	Widely distributed both free and combined as a constituent of various polysaccharides. Forms <i>d</i> -glucosazone.  Obtained synthetically from <i>l</i> -glucosazone. With 2 mol. phenylhydrazine forms <i>l</i> -glucosazone. Obtained synthetically from acrolein. With 2 molecules phenylhydrazine forms <i>l</i> -glucosazone.  Juice of mountain-ash berries. Obtained by condensation of formic aldehyde. Decomposes on heating to 90°-110° C. Obtained by condensation of formic aldehyde.
3. <i>Hexoses of unknown nature and constitution</i> , $\text{C}_6\text{H}_{12}\text{O}_6$ (a) Natural sugars.....	.....	.....	<i>Chondroglucose</i> , from chondrin by treatment with hydrochloric acid. <i>Crocose</i> by hydrolysis of coloring matter of saffron. <i>Eucalyptin</i> , from melitose by hydrolysis. <i>Urine-sugar</i> , from diabetic urine. <i>Hederose</i> , a decomposition product of a glucoside found in the ivy ( <i>Hedera helix</i> ). <i>Indictacin</i> , from the hydrolysis of indican; probably identical with dextrose. <i>Leucose</i> , from the hydrolysis of Chinese green, a coloring matter from a species of <i>Rhamnus</i> . <i>Paragluucose</i> , formed in the vinous fermentation of cane-sugar. <i>Phlorose</i> , from the hydrolysis of phloridzin, <i>Scammonose</i> , from the hydrolysis of a glucoside of scammony. <i>Skimmimose</i> , from the hydrolysis of skimmim, a constituent of a certain Japanese species of rue. <i>Solanose</i> , from hydrolysis of solanin. <i>Tewfikose</i> , forms 5 to 6 per cent of the milk of the Egyptian buffalo cow. <i>Wine sugar</i> , from many natural wines.  <i>β</i> -acrose, only known in form of osazone. Melting-point of osazone 158°-159°. <i>β</i> -formose (pseudoformose, isoformose) obtained by condensation of formic aldehyde. Melting-point of osazone 148° C. Obtained synthetically.
(b) Synthetic sugars.....	.....	.....	
4. <i>Methyl-hexoses</i> <i>α</i> -ribanose..... $\text{CH}_3 \cdot (\text{CHOH})_5 \cdot \text{COH}$	180°	200°	

β-rhamnus-hexose. Digitaflose, $C_7H_{14}O_6$	Sirup	200°	Obtained synthetically. Hydrolysis of the glucoside digitalin.
F. HEPTOSES α-gluco-heptose $CH_2OH \cdot (CHOH)_5CHO$	180°–190°	195°	Obtained synthetically.
β-gluco-heptose $CH_2OH \cdot (CHOH)_5CHO$	Sirup		Obtained synthetically.
d-manno-heptose	134°	200°	Obtained synthetically.
l-manno-heptose	Sirup	203°	Obtained synthetically.
l-manno-heptose	Sirup	210°	Obtained synthetically.
α-gala-heptose		220°	Obtained synthetically.
β-gala-heptose			Obtained synthetically.
β-gala-heptose	Sirup	200°	Obtained synthetically.
G. OCTOSSES. $CH_2 \cdot (CHOH)_6 \cdot CHO$ . α-gluco-octose		210°–212°	Obtained synthetically. mp-point 93°.
β-gluco-octose			Obtained synthetically.
d-manno-octose	Sirup	223°	Obtained synthetically.
Gala-octose			Obtained synthetically.
Rhamno-octose		216°	Obtained synthetically.
H. NONOSSES. $C_9H_{18}O_8$ . α-gluco-nonose	Sirup	230°–233°	Obtained synthetically.
d-gluco-nonose	Sirup 130°	217°	Obtained synthetically.
II. DISACCHARIDES			
A. DERIVATIVES OF PENTOSSES Arabion (di-arabinose) $C_{12}H_{22}O_{11}$			Arabinic acid (metapeptic acid).
B. DERIVATIVES OF HEXOSSES Sucrose	160°		Very widely distributed in the vegetable kingdom. Sucrose, when free from raffinose, is not browned at 120°–135°.
Saccharose, cane-sugar $C_{12}H_{22}O_{11}$	200°		From ergot and other fungi. The hydrate, $C_{12}H_{22}O_{11} + H_2O$ , melts at 130°, becomes anhydrous, solidifies, and again melts at 200°. Forms no osazone.
Trehalose			From the milk of various mammals. The hydrate, $C_{12}H_{22}O_{11} + H_2O$ can be dried at 100° and at 145°–150° begins to lose water and decomposes.
Mycose, trehalblose			From starch by the action of diastase and by the action of ptyalin. The hydrate, $C_{12}H_{22}O_{11} + H_2O$ , loses its water at 100°–105° in vacuo, at 100° to 110° in the air with browning.
Lactose		200°	From malted grain. Begins to decompose at 65°, and melts with marked decomposition at 200°.
Milk sugar, lactobiose			Formed, together with d-fructose, by careful inversion of melitribose.
Maltose		206°	
Maltobiose, ptyalose, cerealose			
Isomaltose		150°–153°	
Melibiose		176°–178°	

TABLE 50—Section A—Continued  
SHOWING THE MORE IMPORTANT CHEMICAL AND PHYSICAL PROPERTIES OF THE CARBOHYDRATES—(COMPILED BY ERVIN E. EWELL)

Name, Synonyms, and Formula	Melting-point °C.	Melting-point of Osazone °C.	Source, etc.
Turanose.....	.....	215°-220°	Formed, together with <i>d</i> -glucose, by careful inversion of melectiose.
Cyclamose.....	.....	.....	From cyclamen tubers.
Agavose.....	.....	.....	From the juice of the agave.
Para-saccharose.....	.....	.....	Formed by the action of a certain yeast plant on cane-sugar in the presence of ammonium phosphate.
C. DERIVATIVES OF HEPTOSES..... $C_{12}H_{22}O_{12}$	.....	.....	Obtained synthetically from a milk-sugar derivative.
III. TRISACCHARIDES $C_{18}H_{34}O_{16}$	.....	.....	Beet-molasses, cotton-seed, eucalyptus manna, etc. Reported by Pellet in cane-molasses.
Raffinose.....	.....	.....	The hydrate ( $C_{18}H_{34}O_{16} + 5H_2O$ ) loses its water on careful heating to 80°; then to 100°-105°; heated quickly it melts below 100°, losing part of its water of crystallization; decomposition begins at 125°-130°.
Meltilrose, gossypose, cotton-sugar.....	.....	.....	Manna of <i>Pinus larix</i> . The hydrate ( $C_{18}H_{34}O_{16} + 2H_2O$ ) loses its water on gradual heating; the anhydrous residue melts at 148°-150°.
Melectiose..... Melectrirose	148°-150°	.....	Tubers of <i>Stachys tubertifera</i> .
Stachyose.....	.....	.....	From the roots of <i>Gentiana lutea</i> .
Gentianose.....	210°	.....	From the roots of <i>Stene vulgaris</i> and other members of the order Caryophyllaceae.
Lactosinose.....	.....	.....	From unripe rye.
Secalose.....	.....	.....	Formed together with <i>d</i> -glucose by the action of diastase on starch at a high temperature.
A sugar of the formula $C_{12}H_{22}O_{14}$ .....	.....	150°-153°	Polysaccharides include soluble starch, dextrin, inulin, the galactans, and a number of plant constituents that are transformed into mono-saccharides by hydrolysis with dilute acids. Very widely distributed in the vegetable kingdom.
IV. POLYSACCHARIDES Starch ( $C_6H_{10}O_5$ )	.....	.....	



TABLE 50—Section B—Continued  
SHOWING THE MORE IMPORTANT CHEMICAL AND PHYSICAL PROPERTIES OF THE CARBOHYDRATES—(COMPILED BY ERVIN E. EWELL)

Name	Specific Rotatory Power, $(\alpha)_D$	Be- havior with Al- kaline Copper Sol.	Behavior with Yeast ( $\alpha$ ) <sub>D</sub>	Oxidation Products	Reduction Products
I. MONOSACCHARIDES					
A. BROSSES					
A. Glycollic aldehyde.....	.....	R.R.T.	.....	First glycollic then oxalic acid.	
B. Trioses		S.R.	Readily fermented	First glycollic then tartaric acid.	
C. Tetroses				First erythritic then tartaric acid.	
C. Erythrose.....	Osazone inactive			{ By gentle oxidation, ara- bonic acid; more vigorous oxid., l-trioxyglutaric ac. d-trioxyglutaric acid.	Arabite
D. Pentoses			Not fermented by yeast.		
1. Aldo-pentoses.....	{ 1104.4° at 18° 2105.4° at 20° 1105.1°	R {	Fermentable by <i>Bacil- lus ethaceticus</i> .	Xylo-trioxyglutaric and trioxybutyric acid.	Xylite Adonite
1. d-arabinose.....	104.1°	R.	.....		
1. d-arabinose.....	Inactive				
1. d-arabinose.....	See foot-note 4.	R.	Not fermented		
1. d-arabinose.....	Inactive				
1. d-arabinose.....	.....				
2. Keto-pentoses					
3. Methyl-pentoses					
Fucose.....	-77°	R.	Unfermentable	l-trioxyglutaric, carbonic, formic, and oxalic acids.	Rhamnite
Rhamnose.....	+8°-9°	S.R.			
Chinovose.....	.....				
E. Hexoses					
1. Aldo-hexoses					
d-glucose.....	See foot-note 6.	P	Fermentable	d-gluconic acid by action of Hr or Cl; by HNO <sub>3</sub> , sac- charic acid.	d-sorbit

\* R. R. T. = reduces at room temperature; R. = reduces; S. R. = strongly reducing; N. R. = no reduction.

1 Scheibler.

2 Lippmann.

3 Rotatory power increases with the concentration; ( $\alpha$ )<sub>D</sub> = 18.425° when c = 9; ( $\alpha$ )<sub>D</sub> = 23.702° when c = 61.7 (Tollens and Schulze).

4 Fischer and Piloty.

5 According to Tollens: 10% sol. of hydrate, ( $\alpha$ )<sub>D</sub><sup>20</sup> = 47.92°; 10% sol. of anhydride, ( $\alpha$ )<sub>D</sub><sup>20</sup> = 52.74°. The rotatory power increaseswith the concentration, and is indicated by the following formulae: For hydrate, ( $\alpha$ )<sub>D</sub><sup>20</sup> = 47.73 + .015534p + .0003883p<sup>2</sup>;For anhydride, ( $\alpha$ )<sub>D</sub><sup>20</sup> = 52.5 + .018796p + .00051683p<sup>2</sup>;

p = per cent d-glucose by weight of the solution.

TABLE 50—Section B—Continued  
SHOWING THE MORE IMPORTANT CHEMICAL AND PHYSICAL PROPERTIES OF THE CARBOHYDRATES—(COMPILED BY ERVIN E. EWELL)

Name	Specific Rotatory Power, ( $\alpha_D$ )	Be- havior with Al- kaline Copper Sol.	Behavior with Yeast	Oxidation Products	Reduction Products
<i>l</i> -glucose.....	-51.4° in 4% sol.	.....	Unfermentable	First <i>l</i> -gluconic then <i>l</i> -sac-	
<i>d</i> -glucose.....	Inactive	.....	Half fermented	charic acid.	
<i>d</i> -mannose.....	12.89°-14.36° at 20°	R.	<i>l</i> -glucose remaining. Fermentable	<i>d</i> -mannonic acid by Br or HNO <sub>3</sub> ; further with HNO <sub>3</sub> , <i>d</i> -manno-saccharic acid.	<i>d</i> -mannite (man- nitol)
<i>l</i> -mannose.....	Leyorotatory	R.	Unfermentable	<i>l</i> -mannonic acid, with Br.	<i>l</i> -mannite
<i>l</i> -mannose.....	Inactive	R.	Is half fermented, the <i>l</i> -mannose remaining	<i>d</i> -saccharic acid.	<i>l</i> -mannite
<i>d</i> -gulose.....	.....	.....	Unfermentable	First <i>l</i> -gulonic then <i>l</i> -sac-	<i>l</i> -sorbitol
<i>l</i> -gulose.....	Weakly dextro- rotatory	.....	Unfermentable	charic acid.	
<i>l</i> -idose.....	.....	.....	.....	First <i>l</i> -idonic then idosac-	
<i>d</i> -galactose.....	See foot-note 1.	R.	Fermentable	charic acid.	Dulcite with other alcohols
<i>l</i> -galactose.....	-73.9° to -74.7°	.....	Unfermentable	With Cl or Br. <i>d</i> -galactonic acid; with HNO <sub>3</sub> , mucic acid.	Dulcite
<i>l</i> -galactose.....	Inactive	.....	<i>l</i> -galactose remaining	First <i>l</i> -galactonic then mucic acid.	
<i>d</i> -talose.....	.....	.....	Unfermentable	First <i>d</i> -talonic then <i>d</i> -talo- mucic acid.	<i>d</i> -talite
Chitose.....	Dextrorotatory	R.	Unfermentable		
2. <i>Keto-Hexoses</i>					
<i>d</i> -fructose.....	See foot-note 2.	R.	Fermentable	A mixture of formic, glycollic and trioxypyruvic acids.	<i>d</i> -mannite and <i>d</i> -sorbitol
<i>l</i> -fructose.....	Dextrorotatory	.....	Unfermentable	.....	<i>l</i> -mannite (acrite)
<i>l</i> -fructose.....	Inactive	R.	Is half fermented, the <i>l</i> -fructose remaining	Aposorbic and other acids	<i>d</i> -sorbitol
Sorbitol.....	-43.4°	R.	Unfermentable		
Formose.....	Inactive	R.	Unfermentable		
Methose.....	.....	R.	Fermentable		
3. <i>Hexoses of unknown na- ture and constitution</i>	See foot-note 3.	.....	.....		
(a) Natural sugars					
(b) Synthetic sugars					
<i>p</i> -formose.....	Inactive	R.	Unfermentable		

4. <i>Methyl-hexoses</i>					
$\alpha$ -rhamno-hexose	— 61.4° at 20°	.....	Unfermentable	Mucic acid	$\alpha$ -rhamno-hexite
$\beta$ -rhamno-hexose	— 79.4° at 28°	.....	.....	<i>l</i> -talomucic acid	
F. <i>Hexoses</i>					
$\alpha$ -gluco-heptose	— 19.7° at 20°	.....	Not readily fermented	.....	$\alpha$ -gluco-heptite
$d$ -manno-heptose	10 gms in 100 cc.	.....	Unfermentable	.....	$d$ -manno-heptite
$l$ -manno-heptose	68.64° at 20°	.....	Unfermentable	.....	$l$ -manno-heptite
$\alpha$ -gala-heptose	.....	.....	.....	Inactive pentoxypimelinic acid.	$\beta$ -manno-heptite
$\beta$ -gala-heptose	.....	.....	.....	Active pentoxypimelinic acid.	
G. <i>Octoses</i>					
Rhamno-heptose	8.4° at 20°	.....	Unfermentable	.....	$\alpha$ -gluco-octite
$\alpha$ -gluco-octose	4—43.9° at 20° 5—50.5° at 20°	.....	Unfermentable	.....	$d$ -manno-octite
$d$ -manno-octose	— 3.3° (ca) at 20°	R.	.....	.....	*
Rhamno-heptose	.....	.....	Unfermentable	.....	$\alpha$ -gluco-nonite
H. <i>Novoses</i>					
$\alpha$ -gluco-nonose	Weakly dextro-rotatory	.....	Unfermentable	.....	
$d$ -manno-nonose	50° (ca) at 20°	.....	Fermentable	.....	
II. <i>DISACCHARIDES</i>					
A. <i>DERIVATIVES OF PENTOSSES</i>					
Arabinose	198.8°	R.	.....	.....	<i>l</i> -arabinose
B. <i>DERIVATIVES OF HEXOSES</i>					
Sucrose	See foot-note 6.	N.R.	Fermentable	Various acids according to conditions.	$d$ -glucose
Trehalose	197.28°	.....	Unfermentable	Forms oxalic acid on heating with dilute HNO <sub>3</sub> .	$d$ -fructose
Lactose	752.53° at 20° for hydrate	R.	No true alcoholic fermentation.	Mucic and other acids according to conditions.	$d$ -glucose and $d$ -galactose

1 According to Rindell and Meissl, when  $t = 10^\circ$  to  $30^\circ$  and  $p = 4.89\%$  to  $35.36\%$ ,  $(\alpha)_D = 83.883 \times 0.785p - .20M$ .  $t = \text{temp.}$   
 2  $(\alpha)_D^{20}$  (25 grams in 100 cc. of solution) =  $-91.8^\circ$ . The specific rotatory power diminishes as the temperature increases, and increases with the concentration.

3 Chondroglucose,  $(\alpha)_D^{20} = -45.8^\circ$ ; crocose, dextrorotatory; eucalyn,  $(\alpha)_D^{20} = 65^\circ$ ; urine-sugar,  $(\alpha)_D^{20} = -26.07$ ; hederose,  $(\alpha)_D^{20} = -98.88^\circ$ ; locaose, inactive; paragalucose,  $(\alpha)_D^{20} = 40^\circ$ ; phlorose,  $(\alpha)_D^{20} = 39^\circ$  to  $41^\circ$ ; scammonose,  $(\alpha)_D^{20} = 17.78^\circ$ ; skimmiose,  $(\alpha)_D^{20} = 24^\circ$ ; solanose,  $(\alpha)_D^{20} = 28.6^\circ$ ; tawfikose,  $(\alpha)_D^{20} = 48.7^\circ$ . All reduce copper except skimmiose (?), and wine-sugar. Eucalyn, hederose, and wine-sugar are unfermentable, and scammonose is fermentable. On oxidation eucalyn yields oxalic acid.

4 Hydrate. 5 Anhydride. 6 Sucrose in 4% solution,  $(\alpha)_D^{20} = 66.8$ ; 70% solution,  $(\alpha)_D^{20} = 65.5^\circ$  (Tollens). 7 Schmoeger.

TABLE 50—Section B—Continued  
SHOWING THE MORE IMPORTANT CHEMICAL AND PHYSICAL PROPERTIES OF THE CARBOHYDRATES—(COMPILED BY ERVIN E. EWELL)

Name	Specific Rotatory Power, ( $\alpha_D$ )	Be-havior with Al-kaline Copper Sol.	Behavior with Yeast	Oxidation Products	Hydrolysis Products
Maltose.....	137° at 20° in 11% sol.	R.	Fermentable	<i>d</i> -saccharic acid on treatment with HNO <sub>3</sub> .	<i>d</i> -glucose
Isomaltose.....	139°-140°	R.	Hydrolysis followed by fermentation.	.....	<i>d</i> -glucose
Melbiose.....	127.3°-139°	R.	.....	.....	{ <i>d</i> -glucose and <i>d</i> -galactose
Turanose.....	65°-68°	R.	Ferments with difficulty or not at all.	.....	<i>d</i> -glucose
Cyclamose.....	-15.15°	.....	.....	.....	Not determined
Agavose.....	Inactive	.....	Unfermentable	.....	Not determined
PARA-SACCHAROSE.....	( $\alpha$ ) <sub>T</sub> = 108°	R.	.....	.....	{ <i>d</i> -galactose and <i>d</i> -glucoheptose
DERIVATIVES OF HEPTOSE: Sugar of the formula C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	.....	.....	.....	.....	.....
III. TRISACCHARIDES					
Raffinose.....	105°-105.7°	N.R.	Fermentable	With HNO <sub>3</sub> , oxalic, saccharic, and mucic acids.	First <i>d</i> -fructose and melbiose; further action decomposes the latter into <i>d</i> -glucose and <i>d</i> -galactose
Melcitose.....	88.65°-88.8° for anhydride	N.R.	Unfermentable	.....	First <i>d</i> -glucose and turanose; further action decomposes the latter into <i>d</i> -glucose, <i>d</i> -galactose, <i>d</i> -glucose, and <i>d</i> -fructose
Stachyose.....	147.9°-148.1° for anhydride.	N.R.	.....	Mucic and other acids.	<i>d</i> -galactose and two other sugars.
Gentianose.....	65.7°	.....	.....	Mucic acid	<i>d</i> -galactose
Lactosinose.....	211.7° at 16°	.....	.....	.....	.....
Scalose.....	-28.6° to -28.9°	.....	.....	.....	.....
A sugar of the formula C <sub>18</sub> H <sub>34</sub> O <sub>16</sub>	143°	R.	Ferments with difficulty or not at all.	.....	<i>d</i> -glucose
IV. POLYSACCHARIDES					
Starch.....	.....	.....	.....	.....	<i>d</i> -glucose, maltose, dextrine, isomaltose, according to conditions.

TABLE 51

INDEX OF SUBSTANCES THAT ARE OR HAVE BEEN USED FOR PURIFYING, DECOLORIZING  
AND CLARIFYING SUGAR-CONTAINING SOLUTIONS

\* Compilation by Prof. Dr. Edmund O. von Lippmann, printed in *Deutsche Zuckerindustrie*, Vol. XXXIV, page 9 (Jan., 1909).—Translation supplied through the courtesy of Dr. Charles A. Browne, Bureau of Chemistry, Washington.

(A list of the abbreviations of references is given at the end of this index.)

I. SULPHUR; ITS ACIDS, COMPOUNDS AND DERIVATIVES

1. Sulphur (Leuchs III, 86; 1836).
  2. Hydrogen Sulphide (Sievier, 1847. in Woodcroft, 94; Hlavati, Chz., 28, 1180).
  3. Hydrogen Persulphide (Hlavati, Chz., 28, 1180).
  4. Sulphuric Acid (Achar, about 1800, Gesch., 407; Kessler, Z., 16, 760; Hagemann, D. Z., 12, 491).
  5. Sulphuric Acid with Lime (Mége, D., 115, 215).
  6. Sulphuric Acid with Zinc Chloride (Thiele, Chz., 20, 404).
  7. Sulphuric Acid with Zinc Sulphate (Terry, 1833, in Woodcroft, 54).
  8. Potassium or Sodium Sulphate (Macfadyen, 1830, Gesch., 423).
  9. Ammonium Sulphate (Dullo, D., 155, 71; Beanes, D., 167, 220).
  10. Sulphuric with Sulphurous Acid (Possoz, D., 170, 64).
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11. Sulphurous Acid (Drapiez, "Bull. de la Société d'encourag.," Paris, 1811, X. 56; Perpere, 1812, and Dubrunfaut, 1829, Zerban, 1908).
  12. Sulphurous Acid and Hydrogen Sulphide (Hlavati, Bl. Ass., 16, 759).
  13. Sulphurous Acid with Calcium Bisulphite (Stolle, D., 114, 305).
  14. Sulphurous Acid with Chloride of Lime and Phenol (Meiner, Bl. Ass., 10, 165).
  15. Sulphurous Acid and Phenol (Kowalski, Z., 55, 396).
  16. Sodium Sulphite (Perrier and Possoz, Z., 12, 128; Rümpler, N.Z., 30, 204).
  17. Potassium Sulphite (Cassel and Kempe, S. ind., 47, 684).
  18. Ammonium Sulphite (Beanes, D., 167, 220).
  19. Calcium Sulphite (Prout, 1810; Melsens, C. r., 55, 729; Calvert, Z., 12, 500).
  20. Barium Sulphite with Oxygen (Bouillant, S. ind., 50, 189).
  21. Magnesium Sulphite (Mehay, Z., 23, 47; Drost and Schulz, Oe., 1885, 891; Degener, D. Z., 24, 203).
  22. Lead Sulphite (Scoffern, 1847, in Woodcroft, 98).
  23. Ferrous Sulphite (Englert and Becker, N. Z., 16, 70).
  24. Aluminum Sulphite (Boulin, 1846, in Zerban, 15; Brandé, 1846, Z., 44, 455; Mehay, Z., 23, 27).
  25. Aluminum Sulphite with Calcium Hydrate (Schubarth, Z., 2, 129).
  26. Aluminum Sulphite with Manganese Sulphate (Massé, Z., 10, 256).
  27. Acid Potassium Sulphite (Z., 1, 254; Cassel and Kempe, S. ind., 47, 684).
  28. Acid Sodium Sulphite (Perrier and Possoz, Z., 12, 128).
  29. Acid Alkali Sulphite with Calcium Bisulphite (Allabard, Engl. Patent No. ?).
  30. Acid Calcium Sulphite (Stolle, 1838, in Zerban, 15; Melsens, D., 117, 136; Reynoso, Z., 12, 500).
  31. Acid Calcium Sulphite with Alum (Leyde, Z., 1, 365).
  32. Acid Calcium Sulphite and Calcium Hydrate and Alum (Lapeyrere, S. ind., 27, 568).
  33. Acid Barium Sulphite, also with Alum (Lapeyrere, see above).
  34. Acid Strontium Sulphite (Melsens, S. ind., 9, 379).
  35. Acid Magnesium Sulphite (Mehay, Z., 23, 26; Hulwa, Oe., 13, 465; Saillard, S. ind., 42, 82).
  36. Acid Iron Sulphite (Becker, N. Z., 16, 6).
  37. Acid Aluminum Sulphite (Stolle, 1838, S. ind., 8, 295; Becker, Z., 35, 924).
  38. Acid Aluminum Sulphite with Aluminum Phosphate (Schiller, Z. B., 12, 509).

39. Calcium Trisulphite (?) (Labarre, Oe., 18, 36).
  40. Basic Magnesium Sulphite (Berggreen, B., 16, 2542).
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41. Hyposulphurous Acid (Talamo, N. Z., 29, 211; Baudry, Z., 53, 260).
  42. Sodium Hyposulphite (Thiele, Chz., 20, 404).
  43. Sodium Hyposulphite with Lime and Aluminum Acetate (D. Z., 33, 912).
  44. Sodium Hyposulphite with Phosphoric Acid or Phosphates (Stein and Crosfield, Z., 53, 1334).
  45. Hyposulphites of the Alkaline Earths and Magnesia (Reece and Price, 1849, in Woodcroft, 106).
  46. Hydrosulphurous Acid (Ranson, Oe., 26, 737).
  47. Ammonium Hydrosulphite (Descamps, S. ind., 65, 673).
  48. Sodium Hydrosulphite (Thiele, Chz., 20, 404; Schiller, Z. B., 22, 683).
  49. Calcium Hydrosulphite with Barium Hydrate (Descamps, S. ind., 65, 673).
  50. Hydrosulphite of Calcium, Barium or Strontium (Descamps, S. ind., 65, 673).
  51. Magnesium Hydrosulphite (Becker, Z., 36, 978).
  52. Cadmium Hydrosulphite (Urbain, S. ind., 50, 31).
  53. Zinc Hydrosulphite (Urbain, see above).
  54. Double Salt of Zinc Hydrosulphite with Sodium Chloride or Bromide and Ammonium Chloride or Fluoride (Harding, S. ind., 66, 742).
  55. Iron or Manganese Hydrosulphite (Descamps, S. ind., 65, 673).
  56. Aluminum Hydrosulphite (Descamps, see above).
  57. Hydrosulphite of Alumina (Becker, Z., 36, 978).
  58. Hydrosulphurous Acid and Phenol (Kowalski, Z., 55, 396).

## II. PHOSPHORUS; ITS ACIDS, COMPOUNDS AND DERIVATIVES

59. Phosphorus Sulphide (Hlavati, Chz., 27, 254).
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60. Phosphoric Acid (Stammer, Z., 9, 433).
  61. Sodium Phosphate (Kuhlmann, Z., 2, 130).
  62. Potassium Phosphate (Blanchard, B., 6, 153).
  63. Ammonium Phosphate (Kuhlmann, Z., 2, 92; Beanes, Amer. patent, 1862).
  64. Sodium Calcium Phosphate (Gwynne, Z., 3, 292).
  65. Calcium Phosphate (Oxland, Z., 2, 130; Ostermann, S. ind., 40, 598).
  66. Barium Phosphate (Heffter, Oe., 22, 71).
  67. Strontium Phosphate (Heffter, see above).
  68. Magnesium Phosphate (Kessler, Z., 15, 525).
  69. Phosphate of Alumina (Oxland, Z., 2, 130).
  70. Acid Ammonium Phosphate (Packert, S. ind., 25, 25).
  71. Acid Ammonium Phosphate with Barium Hydrate (Chameroy, S. ind., 51, 173).
  72. Acid Calcium Phosphate (Richter, 1834, Z., 44, 446; Schott, N. Z., 14, 314).
  73. Acid Calcium Phosphate with Calcium Bisulphite (Barthelémy, S. ind., 52, 468).
  74. Acid Calcium Phosphate with Magnesium Sulphate (Kessler, Z., 15, 51).
  75. Acid Barium Phosphate (Manoury, J. Fabr., 29, 24).
  76. Acid Magnesium Phosphate (Kessler, Z., 15, 51).
  77. Acid Phosphate of Alumina (Oxland, Z., 2, 130).
  78. Calcium Superphosphate (Maguin, J. Fabr., 29, 28).
  79. Superphosphate of Alumina (Daubeny, 1857, in Ling-Roth, 23; Stubbs, Bl. Ass., 9, 912).
  80. Commercial Superphosphate (Casamajor, Z., 34, 1269).
  81. Tribasic Calcium Phosphate, also with Sulphurous Acid (Packert, S. ind., 25, 25).
  82. Tribasic Calcium Phosphate with Alum (Kessler, Z., 15, 51).
  83. Tribasic Calcium Phosphate with Ammonium Phosphate (Lepley, Z., 12, 193).
  84. Tribasic Phosphate of Alumina with Sulphurous Acid (Packert, S. ind., 25, 25).
  85. Manganese Phosphate (Lefranc, S. ind., 53, 410).
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86. Metaphosphoric Acid (Biekmann, S. C., 28, 386; Müller, S. ind., 47, 410).

87. Sodium Calcium Metaphosphate (Gwynne and Young, 1836, in Woodcroft, 59).
88. Sodium Calcium Pyrophosphate (Gwynne and Young, see above).
89. Phosphorous Acid (Hlavati, Chz., 27, 254).
90. Phosphite of Alumina (Spence, Z., 31, 231).
91. Acid Phosphites and Sulphites (Kühnel, Prager Marktb., 1888, 168).
92. Phospho-sulphites of the Alkalies and Alkaline Earths (Prangey and Grobert, S. ind., 54, 425).
93. Hypophosphorous Acid (Hlavati, Chz., 27, 254).

### III. BORON, SILICON, CARBON, THEIR ACIDS, COMPOUNDS AND DERIVATIVES

94. Boric Acid (Payen, 1828, in Weber I, 565).
  95. Boric Acid with Sulphur Powder (Fancher and Clarke, Bl. Ass., 9, 912).
  96. Boric Acid and Borates of the Alkaline Earths (Oppermann, Z., 30, 533; Brear, B., 15, 1224).
  97. Ammonium Borate (Besson, J. Fabr., 43, 1).
  98. Borax (Brear, B., 15, 1224).
  99. Hydrofluoboric Acid (Hlavati, Z., 53, 258).
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100. Silicon Fluoride (Hlavati, Z., 52, 758).
  101. Silicic Acid (Leuchs III, 86, 1836).
  102. Silicic Acid (Kieselguhr), (Heddie, Oe., 16, 441).
  103. Kieselguhr and Saw-dust (Soxhlet, Z., 43, 972).
  104. Hydrated Silicic Acid (Schubarth, Z., 2, 92).
  105. Potassium Silicate, also with Gypsum (Schott, D., 251, 91).
  106. Sodium Silicate (Wagner, Z., 9, 331).
  107. Polysilicates of Magnesium and Aluminum (Hlavati, Chz., 28, 1180).
  108. Zinc Silicate (Hlavati, see above).
  109. Silicate of Alumina, *e.g.*, Brick Dust (Maumené, textbook).
  110. Silicate of Alumina, *e.g.*, Brick Dust, with Caustic Lime (Breyer, Z., 54, 1271).
  111. Hydrofluosilicic Acid (Kessler, Z., 16, 760; Gin, Z., 46, 627; Schoonjans, Chz., 30, 382).
  112. Ammonium Hydrofluosilicate (Mills, N. Z., 39, 115; Whiteman, S. C., 1903, 565).
  113. Ammonium Hydrofluosilicate with Lime (Hlavati, Chz., 28, 1110).
  114. Hydrofluosilicic Acid with Calcium Carbonate (Marix, Bl., 1869, 346).
  115. Magnesium Hydrofluosilicate (Kessler, Z., 16, 760).
  116. Zinc Hydrofluosilicate (Rivière, Bl. Ass., 25, 603).
  117. Lead Hydrofluosilicate (Vivien, Bl. Ass., 8, 24; Sokol, Chz., 21, R., 68).
  118. Basic Lead Salt of Hydrofluosilicic Acid (Hlavati, Chz., 28, 1180).
  119. Aluminum Hydrofluosilicate (Rivière, J. Fabr., 49, 18).
  120. Iron Hydrofluosilicate (Lefranc, Z., 41, 498; Drost Patent, 54, 372).
  121. Hydrofluosilicic Acid with Powdered Iron or Aluminum (Mertens, S. ind., 63, 659).
  122. Manganese Hydrofluosilicate (Kessler, Z., 16, 760).
  123. Hydrofluosilicate of Alumina (Kessler, Z., 15, 525).
  124. Hydrofluosilicic Acid with Alumina (Gin, S. ind., 46, 48).
  125. Carbonic Acid (Barruell, 1811; Oe., 23, 946; Leuchs, 1836, III, 86).
  126. Potassium Carbonate, also with Fuller's Earth (Freund, 1827, Gesch., 369).
  127. Sodium Carbonate (Dubrunfaut, about 1830 (?), Clémandot in Weber, III, 568).
  128. Sodium Potassium Carbonate (Richard, 1856, in Woodcroft, 211).
  129. Ammonium Carbonate (Payen, 1828, in Weber, I, 565; Nind, Z., 1, 595; Stammer, Z., 9, 430).
  130. Acid Sodium Carbonate (Perrier and Possoz, St. J., 1863, 350).
  131. Acid Sodium Carbonate with Alum (Salisbury, Z., 64, 849).
  132. Acid Ammonium Carbonate (Dubrunfaut, about 1830 (?)).
  133. Potassium Percarbonate (Bismar, Oe., 38, 534).

IV. HYDROGEN, OXYGEN, HALOGENS, NITROGEN, THEIR ACIDS, COMPOUNDS  
AND DERIVATIVES

134. Nascent Hydrogen, from Hydroperoxide with Zinc, Lead or Manganese (Manoury, Z., 48, 140).
  135. Hydrogen Peroxide (Frank, Z., 11, 392).
  136. Hydrogen Peroxide with Phosphoric Acid or Alkaline Phosphates (Stein and Crossfield, Oe., 28, 181).
  137. Hydrogen Peroxide with Phosphoric Acid and Magnesia (Pechnik and Bögel in Z., 25, 127).
  138. Hydrogen Peroxide and Bone Black (Ranson, Oe., 26, 737).
  139. Oxygen Gas (Reboux, S. ind., 36, 150; Wayland, S. C., 1893, 611).
  140. Ozonized Air (Schneller and Wisse, S. ind., 39, 467).
  141. Air and Ozonized Air (Steffens, S. ind., 72, 214).
  142. Ozone (Beanes, 1866, in Woodcroft, 392; Lee, B., 2, 64).
  143. Ozone with Chlorine and Soda (Brin, Engl. Patent, 2297).
  144. Ozonized Chlorine (?) (Lewicki, Z., 54, 245).
  145. Ozone with Sulphurous Acid and Barium Hydrate (Verley, S. ind., 53, 301).
  146. Ozone with Chloride of Lime and Alumina (Brin, Engl. Patent, 2297).
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147. Chlorine Gas (Strathing and Smit, 1820, Z., 49, 370; Z., 1, 258; Siemens, 1859, Z., 44, 458; Duncan, St. J., 1882, 274).
  148. Liquefied Chlorine (Reboux, S. ind., 36, 150).
  149. Chlorine with Carbonic Acid (Bismer, Oe., 38, 532).
  150. Chlorine with Acetylene (Carlee, D. Z., 33, 738).
  151. Chlorine with Ethylene (Kitsee, S. C., II, 2, 49).
  152. Hydrochloric Acid (Margueritte, S. ind., 8, 71; Kessler, Z., 16, 761; Erk, Z., 26, 288).
  153. Hydrochloric Acid with Metallic Powders (Hlavati, Z., 52, 758).
  154. Hydrochloric Acid with Alum (Thiele, Chz., 20, 404).
  155. Ammonium Chloride (Macfadyen, 1830, Gesch., 423; Reboux, Z., 84, 94; Licht, St. J., 24, 415).
  156. Potassium Chloride (Macfadyen, 1830, Gesch., 423).
  157. Sodium Chloride (Nash, 1852, in Woodcroft, 151).
  158. Hypochlorous Acid (Z., 1, 255; Bismer, Oe., 34, 532).
  159. Hypochlorous Acid Anhydride (Lagarigue, S. ind., 35, 549).
  160. Hypochlorites of Alkalies (Dobler, S. ind., 66, 517; Hafner, Oe., 37, 86).
  161. Hypochlorites of Alkaline Earths (Herapath, 1862, in Woodcroft, 320).
  162. Hypochlorite of Alumina (used in England about 1880).
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163. Bromine (Maumens, S. ind., 1895, 577).
  164. Hydrofluoric Acid (Frickenhaus, Z., 15, 43; Schoonjans, Chz., 29, 889).
  165. Ammonium Fluoride (Besson, Chz., 27, 863; Bartz, 125).
  166. Ammonium Fluoride with Aluminum (Voss, Z., 50, 438).
  167. Magnesium Fluoride (Kessler, S. ind., 1, 363).
  168. Calcium Fluoride (Kessler, S. ind., 1, 363; Abraham, C. Z., 11, 886).
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169. Nitrous Oxide (Melsens, 1849, D. Z., 25, 1360; Hlavati, Chz., 28, 1180).
  170. Nitrous Acid (Drapiez, in Blachette-Zoega, 1833, 264; Newton, 1849, in Woodcroft, 111).
  171. Nitrites of the Alkalies and Alkaline Earths (Decastro, Z., 29, 270).
  172. Nitric Acid (Kessler, Z., 16, 61).
  173. Calcium Nitrate (Decastro, Z., 29, 270).
  174. Potassium Nitrate (Macfadyen, 1830; Gesch., 423).



## V. ALKALIES, ALKALINE EARTHS, AND THEIR COMPOUNDS

175. Ammonia (Nash, 1852, in Woodcroft, 152; Michaelis, Z., 2, 448).
  176. Ammonia, also with Caustic Lime (Marot, B., 9, 643).
  177. Ammonia with Magnesium or Aluminum Sulphate (Hlavati, S. ind., 65, 673).
  178. Ammonia with Oxalic Acid (Hlavati, Z., 56, 300).
  179. Ammonium Sulphide (Bandris, 1853, in Ling-Roth, 107).
  180. Caustic Potash with Alkali Carbonate (partially causticated plant ash), about 700 in Egypt, Gesch., 134 and 287).
  181. Potassium Sulphide or Sodium Sulphide (Bandris, see 179).
  182. Sodium acetate (Margueritte and Maumené, Z., 28, 845).
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183. Calcium Peroxide (Hlavati, Chz., 27, 254).
  184. Caustic Lime and Hydrated Lime (in Egypt about 700, Gesch., 134 and 287).
  185. Calcium Hydrate with Soda (Beuster, J. Fabr., 32, 2).
  186. Calcium Hydrate with Gypsum (Nathusius, in Bley, 75).
  187. Calcium Chloride (Balling, 1837, Z., 44, 452; Michaelis, Z., 2, 65).
  188. Chloride of Lime (Brandes, 1824, Z., 44, 447, Z., 7, 423).
  189. Chloride of Lime with Sulphurous Acid (Hafner and Bismar, Oe., 37, 199).
  190. Calcium Chloride with Lime or Magnesia (Guignard, Z., 53, 446).
  191. Calcium Carbonate (Maumené, J. Fabr., 17, 22).
  192. Calcium Carbonate with Milk of Lime (Dabrowski, Z., 50, 615).
  193. Calcium Bicarbonate (Reece and Price, 1849, in Woodcroft, 106).
  194. Calcium Nitrate with Sulphate of Alumina (Pape, Chz., 12, 30).
  195. Calcium Sulphate (Howard, 1810, Gesch., 368; Druke, 1816, in Woodcroft, 23; Leyde, Z., 1, 378; Duquesne, D., 196, 83).
  196. Calcium Sulphate with Lime (Kassner, D. Z., 29, 2151).
  197. Calcined Gypsum with Lime (Lelsy, D. Z., 33, 919).
  198. Calcium Acetate (Barth, 1832, Z., 44, 449; Durieux, St. J., 8, 334).
  199. Calcium Borate (Klein, B., 9, 1433).
  200. Calcium Sulphide (Drapiez in Blachette-Zoega, 1833, 264).
  201. Calcium Sulphide with Magnesium Sulphate (Drummond, D., 203, 325).
  202. Calcium Persulphide (Talamo, S. ind., 40, 57).
  203. Calcium Sulphuret (Reece and Price, 1849, in Woodcroft, 106).
  204. Polysulphurets of Calcium or Calcium Sulphide with Ammonia and Sulphurous Acid (Hlavati, S. ind., 72, 487).
  205. Calcium Carbide (Rivière, Bl. Ass., 15, 583).
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206. Barium Oxide Hydrate (Lagrange, J. Fabr., 14, 34; Du Beaufret and Manoury, Z., 40, 590).
  207. Barium Oxide Hydrate with Ammonium Phosphate (Lagrange, J. Fabr., 14, 34).
  208. Barium Oxide Hydrate with Soda (Oppermann, Z., 40, 592).
  209. Barium Oxide Hydrate with Iron Vitriol (Curley, S. ind., 43, 361).
  210. Barium Peroxide (Beaudet, D. Z., 18, 1824).
  211. Barium Peroxide with Phosphoric Acid (Stein and Crossfield, Z., 53, 1334).
  212. Barium Peroxide Hydrate (Ranson, S. ind., 47, 251).
  213. Barium Chloride (Licht, B., 15, 1471).
  214. Barium Chloride with Caustic Soda (Plique, D. Z., 2, 51).
  215. Barium Carbonate (Seyferth, Z., 25, 611; Heffter, Oe., 22, 71; Weisberg, S. ind., 64, 429).
  216. Barium Carbonate with Sodium Phosphate and Sulphurous Acid (Packert, S. ind., 25, 25).
  217. Barium Carbonate with Sulphate of Alumina (Eisenstuck, St. J., 3, 244).
  218. Barium Carbonate with Potassium Permanganate (Talamo, N. Z., 29, 210).
  219. Barium Sulphate with Barium Chloride and Lime (Haesendonck, S. ind., 43, 598).
  220. Barium Sulphide and Sulphuret (Reece and Price, 1849, in Woodcroft, 106; Weisberg, S. ind., 64, 429).
  221. Barium Sulphide with Caustic Soda (Romiguières, S. ind., 26, 682).

222. Barium Sulphide with Magnesium Sulphate (Drummond, D., 203, 325).
  223. Barium Manganate (Lefranc, Bl. Ass., 18, 962).
  224. Barium Silicate (Hlavati, S. ind., 65, 675).
  225. Barium Carbide (Rivière, Bl. Ass., 15, 583).
  226. Barium Carbide with Barium Hydrate (Battistoni, S. ind., 68, 198).
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227. Strontium Oxide (Moureaux, Bl. Ass., 19, 1483).
  228. Strontium Oxide Hydrate (Scheibler, Z., 32, 986).
  229. Strontium Oxide Hydrate with Iron Sulphate (Curely, S. ind., 43, 361).
  230. Strontium Chloride (Kottmann, Z., 32, 899).
  231. Strontium Carbonate (Heffter, Oe., 22, 41).
  232. Strontium Sulphite and Sulphuret (Reece and Price, 1849, in Woodcroft, 106).
  233. Magnesium Alloyed with Potassium, Sodium, Copper, Mercury, Tin, Zinc, or Antimony (Besson, Oe., 36, 466).
  234. Magnesium Powder with Alkalies (Ranson, Chz., 21, 1033).
  235. Magnesium Powder with Acids (Manoury, S. ind., 51, 103).
  236. Magnesium Oxide (Thénard, Z., 13, 128).
  237. Magnesium Oxide Hydrate (Rümpler, D. Z., 4, 52; Oppermann, N. Z., 18, 216).
  238. Magnesium Oxide Hydrate with Sulphurous Acid and Lime (Koebig, S. C., 1894, 274).
  239. Magnesium Oxide Hydrate with Magnesium Carbonate (Rümpler, D. Z., 4, 180).
  240. Magnesium Calcium (Dolomite), Oxide Hydrate (Oppermann and Manoury, S. ind., 1888, 240).
  241. Magnesium Chloride (Nash, 1852, in Woodcroft, 151; Kessler, Z., 16, 760; Z., 23, 74; Drenckmann, D. Z., 17, 1468).
  242. Magnesium Carbonate (Reich, Z., 6, 173; Spreckels, Chz., 28, 1070).
  243. Magnesium Subcarbonate (Stenhouse, 1856, in Woodcroft, 216).
  244. Magnesium Bicarbonate (Reece and Price, 1849, in Woodcroft, 106; N. Z., 25, 91).
  245. Dolomite (Dubreul, B., 6, 155).
  246. Magnesium Sulphate (Bayvet, Z., 10, 256).
  247. Magnesium Sulphate with Lime or Baryta (Manoury, S. ind., 26, 680).
  248. Magnesium Sulphate with Alcohol (Degener, Chz., 12, 174).
  249. Magnesium Sulphide (Dubreul, J. Fabr., 13, 27; Rivière, Bl. Ass., 15, 583).
  250. Magnesium Sulphide and Sulphuret (Reece and Price, 1849, in Woodcroft, 106).
  251. Magnesium Silicate (Hlavati, S. ind., 65, 674).
  252. Magnesium Carbide (Rivière, Bl. Ass., 15, 583).
  253. Radium (?) (C. Z., 1908, 466).

## VI. METALS AND THEIR COMPOUNDS

254. Aluminum Dust (Ranson, Chz., 21, 1033).
255. Aluminum Dust with Alkalies (Ranson, see above).
256. Aluminum Dust with Ammonium Sulphite (Besson, Bl. Ass., 19, 800).
257. Aluminum Dust with Hydrofluoric Acid or Hydrofluosilicic Acid (Mertens, Z., 54, 118).
258. Aluminum Alloys, also with Copper or Zinc Dust (Bessen, Chz., 28, 529).
259. Aluminum Chloride (Nash, 1852, in Woodcroft, 151; Heffter, Oe., 22, 71).
260. Aluminum Chloride with Lime (Siemens, St. J., 18, 256).
261. Aluminum Fluoride (Kessler, Z., 15, 525).
262. Alumina (about 700 in Egypt, Gesch., 135 and 295; Murray, about 1802, Gesch., 368).
263. Hydrate of Alumina (Howard, 1810, Gesch., 368; Z., 2, 92).
264. Colloidal Alumina (Löwig, Z., 29, 905).
265. Fuller's Earth (Frische, Z., 35, 361).
266. Sodium Aluminate also with Sulphurous Acid (Besson, Bl. Ass., 25, 733).
267. Aluminate of the Alkaline Earths (Picque, D. Z., 2, 51).
268. Calcium Aluminate (Oxland, Z., 2, 92).
269. Basic Calcium Aluminate (Guf, Z., 46, 202).

270. Tetra- and Hexa-Basic Aluminate of Calcium or Barium (Gin and Leleux, Bl. Ass., 16, 707).
271. Aluminate of Barium or Strontium (Jacquemart, French Patent, 51, 908, 1861; Rembert, Bl. Ass., 20, 747).
272. Barium Aluminate with Ammonia Alum (Geistodt, Z., 28, 843).
273. Barium Aluminate with Sulphurous Acid (Jaluzot, S. ind., 63, 690).
274. Barium Aluminate with Aluminum Sulphate (Jaluzot, see above).
275. Magnesium Aluminate (used about 1888; Hlavati, S. ind., 65, 674).
276. Sulphite of Alumina (Kessler, Z., 15, 525; Massé, 1860, Z., 44, 458).
277. Sulphate of Alumina with Phosphoric Acid (Stein and Crosfield, Oe., 28, 183).
278. Basic Sulphate of Alumina (Hunt, Z., 30, 361; Brünjes, D. Z., 25, 19).
279. Alum (about 700 in Egypt, Gesch., 135; Hermbstaedt, "Anleit. z. Fabrik. des Zuckers," Berlin, 1811, 86).
280. Alum with Sodium Carbonate (Salisbury, Z., 54, 1274).
281. Alum with Lime and Alcohol (Derosne, Oe., 23, 948).
282. Alum also with Sulphate of Alumina (Howard, 1812, Z., 44, 446).
283. Aluminum Acetate (Oxland, 1850, in Ling-Roth, 121; Schubarth, Z., 2, 92).
284. Tartrate of Alumina (Dumas, C. Z., 1906, 939).
285. Oxalate of Alumina (Sievier, 1847, in Woodcroft, 94; Mialhe, D., 99, 482; Dumas, C. Z., 1906, 939).
286. Aluminum Phosphate (Oxland, Z., 2, 92, and 2, 130).
287. Aluminum Silicate (Maumené, Lehrbuch).
288. Aluminate Silicates (Gans, Z., 57, 206).
289. Iron- and Quartz-containing Clay (Harm, D. Z., 22, 1104).
290. Aluminum Sulphide (Hlavati, Chz., 27, 254).
291. Ferrous Oxide (Hills, 1850, in Woodcroft, 121).
292. Iron Hydroxide, also with Gypsum (Rousseau, Z., 11, 671).
293. Iron Sesquioxide, also with Ozone (Wayland, Chz., 19, 1519).
294. Iron Sesquioxide Hydrate (Wackernie, S. ind., 47, 215).
295. Iron Peroxide (Reynolds, 1859, in Woodcroft, 250).
296. Iron Ochre (Martineau, 1815, in Woodcroft, 21).
297. Iron Chloride (Sievier, 1847, in Woodcroft, 94; Karl, Z., 18, 317; Licht, N. Z., 11, 63).
298. Ferrous Chloride (Maumené, S. ind., 1895, 577).
299. Iron Oxy-chloride (Spunt and Schachtrupp, N. Z., 30, 216).
300. Ferrous Fluoride (Junius and Gouthiere, Chz., 25, 603).
301. Iron Carbonate (Reynolds, 1859, in Woodcroft, 250).
302. Ferric Sulphate (Sievier, 1847, in Woodcroft, 94; Karl, Z., 18, 317).
303. Basic Ferric Sulphate (Mehrie, Z., 32, 385).
304. Ferrous Sulphate (Bayvet, Z., 10, 256; Mehrie, Z., 32, 385).
305. Iron Vitriol with Alkaline Earths (Curely, S. ind., 43, 361).
306. Iron Vitriol with Barium Hydrate (Beaufret, Bl. Ass., 10, 803).
307. Iron Vitriol with Gypsum (Lohmann, 1817, Z., 44, 447).
308. Iron Vitriol with Zinc (Schetke, Chz., 1906, 23).
309. Iron Vitriol with Albuminates (Karl, Z., 18, 317).
310. Iron Nitrate (Sievier, 1847, in Woodcroft, 94).
311. Salts of Ferric Acid, so-called "Ferrites" (Liesenberg, about 1892).
312. Iron Cyanide and Sulphurous Acid (Thompson, Z., 50, 957).
313. Potassium Ferrocyanide (Sievier, 1847, in Woodcroft, 94).
314. Potassium Ferrocyanide, also with Sulphurous Acid (Boot, Java Archiv., 1903, 1046).
315. Calcium Ferrocyanide (Therry, 1833, in Woodcroft, 54).
316. Chromium Peroxide (Piettre, Bl. Ass., 19, 1381).
317. Chromic Acid and Salts of Chromic Acid (Maumené, S. ind., 1895, 57).
318. Acid Chromic Acid Salts (Maumené, see above).
319. Chromium Sulphate (Lefranc, S. ind., 58, 410).
320. Chromium Phosphate (Lefranc, see above).

321. Manganese Dust with Acids (Manoury, S. ind., 51, 103).
  322. Manganese Oxide (Spreckels, Chz., 28, 1270).
  323. Manganous Oxide (Eachran, D., 251, 91).
  324. Manganese Dioxide (about 1836, Bley, 47; Frickenhaus, Z., 10, 301; Piettre, Bl. Ass., 19, 1351).
  325. Manganese Chloride (Manoury, about 1880).
  326. Manganese Chloride with Oxalic Acid (Fontenille, S. ind., 54, 425).
  327. Manganese Carbonate (Newton, 1859, in Woodcroft, 253).
  328. Manganese Sulphate (Massé, Z., 10, 256).
  329. Manganates of the Alkalies and Alkaline Earths (Hawes, 1853, in Woodcroft, 163).
  330. Sodium Manganate (Knaggs, 1866, in Woodcroft, 384).
  331. Manganate of Lime (Z., 1, 256; Lefranc, Bl. Ass., 18, 962).
  332. Potassium Permanganate (Maumené, J. Fabr., 1894, 51).
  333. Sodium Permanganate (Knaggs, 1866, in Woodcroft, 384).
  334. Calcium Permanganate (Fayolle, S. ind., 52, 554).
  335. Aluminum Permanganate (Fayolle, see above).
  336. Permanganates with Barium Carbonate and Oxalic Acid (Talamo, N. Z., 29, 210).
  337. Copper Sulphate with Lime (Hlavati, Z., 56, 300).
  338. Lead Dust, also with Acids (Manoury, S. ind., 51, 103).
  339. Lead with Sulphides of the Alkalies (Bandris, Ling-Roth, 107).
  340. Lead Oxide (about 1836, Bley, 126).
  341. Plumbic Hydrate (Gwynne, Z., 3, 392; Lagrange, S. ind., 1892, 468; Wohl and Kollrepp, Z., 55, 60).
  342. Litharge (Pfeifer and Langen, N. Z., 19, 131).
  343. Lead Peroxide (Maumené, S. ind., 1895, 577; Piettre, Bl. Ass., 19, 1351).
  344. Plumbites of the Alkaline Earths (Galloway, 1852, in Woodcroft, 147).
  345. Lead Carbonate (Hills, 1850, in Woodcroft, 121; Besson, Chz., 28, 1270).
  346. Lead Sulphate (Scoffern, 1850, in Woodcroft, 115).
  347. Lead Nitrate (Lagrange, S. ind., 1892, 468).
  348. Lead Nitrate with Sulphate of Alumina (Pape, Chz., 12, 30).
  349. Basic Lead Nitrate (Wohl and Kollrepp, Z., 55, 60).
  350. Lead Acetate (Scoffern, 1847, in Zerban, 15; Gwynne, Z., 3, 392).
  351. Lead Subacetate, also with Sodium Sulphide (Maumené).
  352. Lead Subacetate with Chalk (Pajot de Charmes, 1821, Gesch., 369).
  353. Lead Subacetate with Sulphurous Acid (Scoffern, D., 117, 265; Ling-Roth, 81 and 82).
  354. Lead Triacetate (?) (Gwynne and Young, in Woodcroft, 59).
  355. Lead Saccharate (Gwynne, 1850, in Woodcroft, 116; Wohl and Kollrepp, Z., 54, 854).
  356. Lead Albuminate (Gwynne and Young, 1836, in Woodcroft, 59).
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357. Zinc Dust with Mineral Acids (Manoury, S. ind., 51, 103).
  358. Zinc Dust with Sulphuric Acid and Barium Sulphide (Cripò, St. J., 24, 416).
  359. Zinc Dust with Sulphurous Acid, also with Ferrocyanides (Boot, Oe., 27, 717).
  360. Zinc Dust with Hydrofluoric Acid (Mertens, Z., 54, 118).
  361. Zinc Dust with Tartaric Acid (Koperski, Z., 54, 1271).
  362. Zinc Dust with Alkalies (Ranson, Chz., 21, 1033).
  363. Zinc Dust with Dolomite (Hlavati, Bl. Ass., 16, 759).
  364. Zinc Dust with Ammonium Sulphide (Brünn, Chz., 31, R., 459).
  365. Coppered Zinc-Powder (Verley, Chz., 24, 596).
  366. Zinc Iron Alloys (Mertens, Z., 54, 118).
  367. Zinc Chloride (Gauchy, N. Z., 13, 43; Heffter, Oe., 22, 71).
  368. Zinc Fluoride (Hlavati, Z., 53, 258).
  369. Zinc Oxide (about 1836, Bley, 126).
  370. Zinc Hydrate (Wilson, 1815, Gesch., 368).
  371. Zinc Hydrocarbonate (Perrin, Chz., 22, 54; Mittelstaedt, D. Z., 23, 1112).
  372. Zinc Hydrocarbonate with Oxalic Acid (Moureaux, Bl. Ass., 19, 1483).

373. Zinc Sulphate (Wilson, 1818, in Woodcroft, 27; Z., 44, 447; Hermbstädt, in Weber, 1829, 100).
374. Zinc Sulphate with Barium Hydrate (Wackernie, S. ind., 53, 201, 61, 718).
375. Zinc Nitrate (Decastro, St. J., 19, 340).
376. Zinc Nitrate with Alkali Sulphide (Decastro, see No. 375).
377. Zinc Nitrate with Calcium Sulphide or Barium Sulphide (Decastro, see No. 375).
378. Zinc Aluminate (Hlavati, S. ind., 65, 674).
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379. Cadmium Oxide (Mouraux, Bl. Ass., 10, 1483).
380. Cadmium Carbonate (Mouraux, see No. 379).
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381. Tin Dust (Besson, Chz., 27, 863).
382. Stannic Oxide, also with Soda (B., 19, R., 520).
383. Stannic Hydrate (Wilson, 1815, Gesch., 388).
384. Stannic Chloride (Nash, 1852, in Woodcroft, 151; Maumené, J. Fabr., 20, 7).
385. Stannous Chloride (Nash, 1852, see No. 384; Havemeyer, 1869, in Zerban, 77; Manoury, Z., 34, 1275; Maumené; S. ind., 1895, 577).
386. Stannous Chloride with Sulphuric Acid (Thiele, Chz., 20, 404).
387. Stannous Nitrochloride (Nash, 1852, see No. 384).
388. Stannous Fluoride (Ranson, Chz., 24, 1026).
389. Stannic Sulphate (Anderson, 1856, in Woodcroft, 218).
390. Stannous Sulphate (Oe., 15, 76).
391. Tin Nitrate (Reynolds, 1859, in Woodcroft, 250).
392. Tin Chloronitrate (Reynolds, 1859, see No. 391).
393. Stannic Acid or Metastannic Acid (Reynolds, 1859, see No. 391).
394. Stannates of the Alkalies (Reynolds, 1859, see No. 391).
395. Stannates of the Alkaline Earths (Reynolds, 1859, see No. 391).
396. Metastannates of the Alkalies and Alkaline Earths (Reynolds, 1859, see No. 391).
397. Aluminum Metastannate (Reynolds, 1859, see No. 391).
398. Mercury Peroxide (Piettre, Bl. Ass., 19, 1351).
399. Mercuric Nitrate (S. C., II, 4, 216).
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400. Antimony Dust (Besson, Chz., 27, 863).
401. Antimony Tin Alloy (Mertens, S. ind., 63, 659).
402. Antimony Peroxide (Piettre, see No. 398).
403. Antimony Sulphide (about 1670, Gesch., 314).
404. Bismuth Nitrate (Sievler, 1847, in Woodcroft, 94).
405. Bismuth Salts (Hawes, 1853, in Woodcroft, 163).
406. Ammonium Molybdate (Wichardt, D. Z., 31, 652).
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407. Salts of Tungstic Acid (Reynolds, 1859, in Woodcroft, 259).
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408. Titanic Acid (Employed in England about 1880).
409. Ferrititanite (Liesenberg, about 1892).
410. Thorium- and Monazite-Earths (Browne, C. Z., 1904, 568).

VII. ORGANIC SUBSTANCES AND COMPOUNDS; BONE BLACK AND ITS SUBSTITUTES

411. Extract of Gall-Apples (about 700 in Egypt, Gesch., 135).
412. Tannins and Tanning Liquors (Dorion, 1816, Z., 49, 578; Leuchs, 1836; III. 86; Luther, Chz., 29, 1091).
413. Quebracho, Valonea, and Sumach (Hlavati, Chz., 27, 254).
414. Tannic Acid (Wagner, Z., 9, 331; Walkhoff, 1863, Z., 44, 459).
415. Liquid Tannic Acid (?) (Elias, S. ind., 1895, 20).
416. Tannate of Potassium or Ammonium (Galloway, 1853, in Woodcroft, 171).
417. Tannic Acid with Lime (about 1836, Bley, 126; Heffter, Oe., 16, 442).
418. Tannic Acid with Salts of Barium or Strontium (Heffter, see No. 417).

419. Tannic Acid with Alumina (Heffter, see No. 417).
420. Tannic Acid with Tartaric Acid, Metaphosphoric Acid, and Hydrofluosilicic Acid (Royers, S. ind., 50, 32).
421. Tannic Acid with Glue, Starch or Albumen (Heffter, see No. 417).
422. Pertannic Acid (?) (Meritens, Z., 28, 800).
423. Gallic Acid (Royers, see No. 420; Kowalski, Chz., 26, 972).
424. Gallate of Potassium or Ammonium (Galloway, 1853, see No. 416).
425. Acetic Acid, also with Sulphurous Acid (Z., 20, 741; Stutzer and Wernekinck, S. ind., 51, 114). For acetates look under the list metals.
426. Wood Vinegar (Leidenfrost, Z., 20, 746).
427. Butyric Sulphonic Acid (?) (Spreckels, Chz., 28, 1270).
428. Fatty Acids with Sulphurous or Sulphuric Acid (Spreckels, Z. ang., 1902, 891; Chz., 28, 1072).
429. Stearic Acid (Z., 2, 91; Wagner, Z., 9, 331).
430. Ammonium Stearate (Besson, Chz., 27, 863).
431. Stearic Acid with Sulphites of the Alkalies or Magnesium (Stewart, Z., 57, 268).
432. Stearic-Sulphonic Acid (Spreckels, see No. 427).
433. Palmitic-Sulphonic Acid (Spreckels, see No. 427).
434. Margaric Acid (Pidding, 1853, in Woodcroft, 162).
435. Oleic Acid (Pidding, see No. 434; Thénard, Z., 8, 130).
436. Oleic-Sulphonic Acid (Spreckels, see No. 427).
437. Oxalic Acid (Leuchs, 1836, III, 86; Wagner, Z., 9, 331; Eissfeldt, Z., 21, 1102).
438. Oxalic Acid with Ammonia, Magnesium and Zinc (Besson, Bl. Ass., 18, 616).
439. Ammonium Oxalate (Sievier, 1847, in Woodcroft, 94; Besson, J. Fabr., 43, 1).
440. Oxalic Acid with Barium Carbonate and Permanganate (Talamo, N. Z., 29, 210).
441. Tartaric Acid (Possos, Z., 23, 27; Stutzer and Wernekinck, S. ind., 51, 114).
442. Ammonium Tartrate (Besson, Chz., 27, 863).
443. Malic Acid with Metallic Bases or Carbonates (Moreaux, Bl. Ass., 19, 1483).
444. Citric Acid with Metallic Bases or Carbonates (Moreaux, Bl. Ass., 19, 1483).
445. Citric Acid, also with Polysilicates (Hlavati, Chz., 28, 1180).
446. Salicylic Acid (Hulwa, Z., 25, 640; D. Z. 9, 7).
447. Resin Acids (Leuchs, 1836; III, 86).
448. Pimaric Acid (Schiller, Z. B., 12, 33).
449. Pectic Acid (Acar in Wagner's Technologie, 12th Ed., 563).
450. Formaldehyde (Boulet, Chz., 20, 12; Friedrich, Chz., 27, 1183; Simpson, Bl. Ass., 25, 531).
451. Acetaldehyde (Newton, 1849, in Woodcroft, 111; Melsens, 1849, D. Z., 25, 1306; Boulet, Chz., 20, 12).
452. Methylalcohol (Trovach, D. Z., 11, 1302).
453. Alcohol (Jennings, 1825, in Woodcroft, 33; Pesier, Z., 11, 522).
454. Alcohol with Chlorine Gas (Duncan, St. J., 22, 274).
455. Alcohol with Acetic Acid (Paulet, 1837; Z., 14, 641; 19, 376).
456. Alcohol with Hydrochloric Acid, Nitric Acid or Sulphuric Acid (Ure, 1830, in Woodcroft, 49).
457. Alcohol with Sulphuric Acid and Gypsum (Duquesne, D., 196, 83).
458. Alcohol with Sulphurous Acid (Stolle, D., 114, 305).
459. Alcohol with Alum and Lime (Derosne, 1810, Oe., 23, 948).
460. Alcohol with Magnesium Sulphite (Degener, Chz., 12, 174).
461. Rum or Gin (Stokes, in Weber, III, 236).
462. Glycerine (Rabe, Z., 14, 124).
463. Glucose and its Salts (?) (Bielmann, S. C., 28, 386).
464. Saccharites of Lead or of the Alkaline Earths (Reece and Price, 1849, in Woodcroft, 106; Gwynne, Z., 3, 392; Stammer, Z., 12, 336).
465. Magnesium Saccharate (Galloway, Z., 4, 81).
466. Starch with Caustic Lime (Steinkamp, 1848, in Woodcroft, 102).
467. Hydrocarbons and Petroleum (Carbonelle, S. ind., 33, 455).
468. Kerosene with Alumina and Metallic Powder (Z., 53, 444).
469. Kerosene Oil (Spreckels and Kern, Z., 53, 378).
470. Tar Oils (Newton, 1849, in Woodcroft, 111).

471. Benzol or Toluol, also with Sulphurous Acid or Hydrosulphurous Acid (Kowalski, Z., 52, 909).
  472. Phenol (Fishman, Z., 21, 313; D. Z., 21, 9, 7).
  473. Phenol with Benzol or Petroleum (Kowalski, Z., 55, 396).
  474. Phenol with Chloride of Lime (Menier, Bl. Ass., 10, 165).
  475. Oxybenzol (Kowalski, see No. 471).
  476. Oxynaphthalin and Oxyanthracene, also with Sulphurous Acid or Hydrosulphurous Acid (Kowalski, see No. 471).
  477. Oxyanthraquinone (Kowalski, see No. 473).
  478. Fats with Sulphurous or Sulphuric Acid (Spreckels, Z. ang., 1902, 891; Chz., 28, 1072).
  479. Tallow or Lard with Sulphurous or Sulphuric Acid (Spreckels & Kern, Z., 55, 571; 53, 878).
  480. Fatty Oils and Mineral Oils (Bouvier, Z. B., 1896, 386).
  481. Fatty Oils with Sulphurous or Sulphuric Acid (Spreckels, Z. ang., 1902, 891).
  482. Fatty Oils with Soda (Brooman, 1857, in Woodcroft, 232).
  483. Wax and Neutral Fat (Leuchs, III, 86).
  484. Spermaceti and Spermaceti Oil (Pidding, 1853, in Woodcroft, 162).
  485. Stearine and Palmatine (Carlee, D. Z., 33, 738).
  486. Fish Oil with Sulphurous or Sulphuric Acid (Spreckels, Z., 55, 571; Z. ang., 1902, 891).
  487. Linseed Oil with Sulphuric Acid (Spreckels and Kern, Z., 53, 878).
  488. Castor Oil with Sulphuric Acid (Spreckels, Z., 55, 571).
  489. Soap (Basset, Z., 7, 381).
  490. Ammonia Soaps (Brooman, Z., 8, 449; Besson, J. Fabr., 43, 1).
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491. Turpentine (Newton, 1849, in Woodcroft, 111; Carlee, D. Z., 33, 738).
  492. Turpentine and Sulphuric Acid (Spreckels and Kern, Z., 53, 878; 55, 571).
  493. Tar with Sulphurous or Sulphuric Acid (Spreckels, Z. ang., 1902, 891).
  494. Tar Oil (Pidding, 1853, in Woodcroft, 162).
  495. Tar Oil also with Alumina and Metallic Powders (Z., 53, 444).
  496. Tar Oils with Sulphurous or Sulphuric Acid (Spreckels, Z. ang., 1902, 891).
  497. Resin (Pidding, 1853, in Woodcroft, 162).
  498. Resin and Sulphuric Acid (Spreckels and Kern, Z., 53, 878).
  499. Pitch (Pidding, 1853, see No. 497).
  500. Creosote (Newton, 1849, in Woodcroft, 111).
  501. Shellac (Greiger, S. ind., 54, 23).
  502. Carbon Bisulphide (Cklandi, S. ind., 25, 268).
  503. Mustard Oil (Leuchs, 1836, III, 86; Newton, 1842, in Woodcroft, 111).
  504. Radish Oil (Newton, 1842, see No. 503).
  505. Sulphur-containing Ethereal Oils (Spreckels, Chz., 29, 1307).
  506. Ethereal Oils with Sulphurous or Sulphuric Acid (Spreckels, Z. ang., 1902, 891; Chz., 28, 1072).
  507. Animal Oil (Pidding, 1853, in Woodcroft, 162).
  508. Eucalyptol (Fry, Amer. Patent, No. 472,989).
  509. Eucalyptus Oil and Sulphuric Acid (Spreckels, Z., 55, 571).
  510. Indigo White (Bielmann, S. C., 28, 386).
  511. Milk (about 600, in Persia, Gesch., 102; Batley, 1810, Gesch., 369; Hermstadt, 1811).
  512. Casein (Krüger, Z., 9, 221).
  513. Albumen (about 700, in Egypt, Gesch., 135 and 209; Wilcox, Bl. Ass., 9, 912).
  514. Calcium Albuminate (Karl, Z., 18, 317).
  515. Calcium Albuminate with Iron Vitriol (Karl, see No. 514).
  516. Blood (about 1700, Gesch., 324).
  517. Hay or grass (Hlavati, Chz., 27, 254).
  518. Bark of Trees, as Elm Bark (Stokes, in Weber, III, 236).
  519. Cork (Wagner, Z., 43, 630).
  520. Plane Shavings (Leuchs, 1836; III, 86).
  521. Wood Dust (Wiechmann, 1885; D. Z., 28, 1544).

522. Sawdust (Hills, 1853, in Woodcroft, 163; Casamajor, 34, 1269).
523. Wood Meal from Mechanical Pulp Process (Soxhlet, Z., 43, 972).
524. Wood Wool (Excelsior) (Müller and Schubert, Z., 44, 233).
525. Wood Pulp or Paper Pulp (Spreckels, Chz., 28, 1270).
526. Linen or Cotton Fabrics and Threads (Ost. Z., 58, 556).
527. Bran (Tyre, Z., 50, 475).
528. Dead Yeast (Clowes, Z., 54, 1286).
529. Yeast with Hydrochloric Acid (Effront, Z., 58, 326).
530. Peat with Calcium Sulphite (Nowak, Z., 53, 988).
531. Peat-Coal or Lignite (Maumené, Z., 4, 452).
532. Calcium Humate (Schmidt and Degener, D. Z., 20, 209).
533. Brown Coal (Böttcher, 1836, Gesch., 377).
534. Brown Coal Coke (Knauer, Z., 11, 350).
535. Wood Charcoal (Lowitz, 1793, Gesch., 368).
536. Charcoal Dust (Remmers, Z., 35, 369).
537. Coal-Dust, also with Alumina (Pajot de Charmes, 1821, Gesch., 369).
538. Lamp-Black (Martineau, 1815, in Woodcroft, 21).
539. Sugar Charcoal (Sievler, 1847, in Woodcroft, 94).
540. Plant-Blood Charcoal (Degener, Z., 46, 492).
541. Bituminous Coal (Martineau, 1815, in Woodcroft, 21; Payen, 1830, Z., 49, 594).
542. Carbon-Alumina (?) (Kachmarkiewicz, C. Z., 1906, 229).
543. Alumina Impregnated with Carbonized Blood (Olschewsky, Z., 32, 525).
544. Carbonized Kieselguhr (Infusorial Earth) (Heddle, Z., 37, 478).
545. Kieselguhr Impregnated with Carbonized Fatty Residues (about 1900, in America).
546. Bone Black (Figuier and Magnes, Gesch., 368).
547. Bone Black with Hydrogen Peroxide (Mastbaum, Z., 37, 704).
548. Bone Black Saturated with Carbonic Acid or Sulphurous Acid (Lach, Z., 46, 497).
549. Bone Meal (Hills, 1853, in Woodcroft, 163).
550. Osteine (Brunon and Rothé; Z., 54, 848).
551. Ferrocyanide Residues (So-called Coal-settlings) (Cavaillon, 1817, in Woodcroft, 25; Gawalowski, Oe., 18, 718).
552. Powder-Settlings of Stearin Factories (Lach and Benies, S. ind., 1895, 20).
553. Graphite with Bone Black and Zinc Bloom (Macherski and Koperski, Z., 57, 1121).
554. Graphite with Sand and Zinc Powder (Macherski and Koperski, Z., 57, 1044).
555. Anthracite (Hlavati, Z., 56, 300).
556. Coal-Tar with Lime (Lemaire, S. ind., 9, 56).
557. Carbonized Scums (Karlik, Oe., 32, 256).
558. Gravel (Bergmann, 1840, Z., 29, 1184; Meyer, 1879, Z., 30, 1149).
559. Bauxite (Hlavati, Z., 56, 300).
560. Calcined Phosphate-Slag (Lachaux, S. ind., 50, 677).
561. Cement (Harm, D. Z., 25, 1946).
562. Brick-Dust with Lime (Breyer, S. ind., 65, 655).
563. Pumice Stone (Saunders, 1835, in Woodcroft, 56).
564. Talc or Meerscham (Hlavati Chz., 28, 1180).
565. Mica (Hlavati, Chz., 28, 1180).
566. Natural Zeolite (Riedel, S. ind., 70, 230).
567. Permutite = Artificial Zeolite (Riedel, see No. 566).
568. Soil from the Beet Storehouse (Kohlrausch, Z., 28, 215).

## VIII. ELECTROLYTIC SUBSTANCES

569. Hydrogen (Kugler, Chz., 32; R., 454).
570. Ozone (Schollmeyer, Chz., 24, 825).
571. Chlorine, Bromine, Iodine, Fluorine (Spillern-Spitzer, Z., 53, 244).
572. Sulphurous Acid (Lallement, S. ind., 53, 301).



573. Sulphurous Acid or Sulphites with Lead, Zinc, Aluminum, Iron, or Tin (Baudry and Charitonenko, Z., 50, 625).
574. Hydrosulphurous Acid (Ranson, Oe., 26, 737).
575. Coal (Despeissis, Battut, Z., 46, 624).
576. Wood Charcoal (Hlavati, Z., 53, 258).
577. Alkaline Earths (Gin and Lelux, Z., 53, 627).
578. Calcium Carbonate (Schwerin, D. Z., 29, 451).
579. Barium Salts (Bonillaut, S. ind., 50, 189).
580. Barium Aluminate (Rembert, Bl. Ass., 20, 966).
581. Magnesium (Urbain, Bl. Ass., 16, 719).
582. Magnalium (Murphy, J. Fabr., 44, 18).
583. Magnesium Hydroxide (Schwerin, Chz., 28, 626).
584. Magnesium Carbonate (Schwerin, D. Z., 29, 451).
585. Zinc (Schollmeyer, Z., 46, 624).
586. Zinc Alloy with Calcium or Antimony (Hlavati, Z., 53, 258).
587. Basic Zinc Salts (Wohl and Kollrepp, D. Z., 27, 1280).
588. Cadmium (Urbain, Bl. Ass., 16, 719).
589. Lead (Javaux, Gallois, Dupont, Z., 46, 626).
590. Lead-Antimony Alloy also with Manganese Sulphate (Piettre and Nodon, D. Z., 27, 1211).
591. Lead Oxides (Z., 46, 626).
592. Lead Peroxide (Piettre and Nodon, Bl. Ass., 19, 1351).
593. Lead Saccharate (Wohl and Kollrepp, D. Z., 27, 1280).
594. Basic Lead Salts (Wohl and Kollrepp, see No. 593).
595. Aluminum (Z., 46, 626).
596. Aluminum-Magnesium (Browne, Z. ang., 1908, 174).
597. Aluminum-Manganate with Zinc Hydroxide or Iron Hydroxide (Delavierre, Z., 53, 1106).
598. Alumina (Z., 46, 626).
599. Iron (Jennings, 1846, Clement, 1848, in Woodcroft, 89 and 103; Malgrot, Z., 46, 625).
600. Iron Bisulphide (Aschermann, Chz., 26, 683).
601. Manganese-Silicon Alloy (Hlavati, Z., 53, 258).
602. Manganese Dioxide (Hlavati, Z., 53, 626).
603. Hydrated Manganese Peroxide (Piettre and Nodon, Bl. Ass., 19, 1351).
604. Manganates of the Alkalies and Alkaline Earths (Lavollay and Bourgoin, D. Z., 25, 330).
605. Chromium Peroxide (Piettre and Nodon, see No. 603).
606. Nickel (Horsin-Déon, Oe., 28, 162).
607. Nickel with Sulphurous Acid or Sulphites (Baudry and Charitonenko, Z., 50, 625).
608. Copper (Görz, Z., 46, 624).
609. Iron (Horsin-Déon, Bl. Ass., 16, 729).
610. Antimony (Piettre and Nodon, D. Z., 27, 1211).
611. Antimony Peroxide (Piettre and Nodon, Bl. Ass., 19, 1351).
612. Mercury (Polaczek, Bl. Ass., 16, 720; Gurwitsch, Z., 54, 1030).
613. Mercury Amalgams (Polaczek, see No. 612).
614. Mercury Peroxide (Piettre and Nodon, Bl. Ass., 19, 1351).
615. Easily-Fluid Mercury Alloys (Palms, Bl. Ass., 17, 274).
616. Silver (Horsin Déon, Oe., 28, 162).
617. Silver with Sulphurous Acid or Sulphites (Baudry and Charitonenko, Z., 50, 625).
618. Platinum (Collette, Z., 46, 623; Thomas and Howe, S. ind., 66, 624).
619. Platinum Antimony Alloy, also with Manganese Sulphate (Piettre and Nodon, D. Z., 27, 1211).
620. Platinized Copper (Charitonenko, S. ind., 53, 272).
621. Sebonaft = "Solid Mineral Oil" (Nowakowski, C. Z., 17, 277).
622. Straw-Meal (Lenze, D. Z., 33, 937).

## ABBREVIATIONS OF REFERENCES

<i>Abbreviations</i>	<i>Reference</i>
Bartz	Claassen-Bartz's "Zuckerfabrikation" (Leipzig, 1905).
B	Berichte der deutschen chemischen Gesellschaft (R = Referate).
Bl.	Bulletin de la Société chimique.
Blanchette Zoega	"Manuel du fabricant et du raffineur de sucre" (Paris, 1833).
Bley	Bley's "Zuckerbereitung aus Runkelrüben" (Halle, 1836).
Bl. Ass.	Bulletin de l'association des chimistes.
Chz.	Chemiker-Zeitung (R = Repertorium).
C. r.	Comptes rendus.
C. Z.	Centralblatt für die Zuckerindustrie.
D.	Dingler's polytechnisches Journal.
D. Z.	Die Deutsche Zuckerindustrie.
Gesch.	Lippman's "Geschichte des Zuckers" (Leipzig, 1890).
J. fabr.	Journal des fabricants de sucre.
Ling-Roth	Ling-Roth's "Guide to the Literature of Sugar" (London, 1890).
Leuchs	Leuchs "10,000 Erfindungen und Ansichten" (Nürnberg, 1871).
Maumené	Maumené's "Traité de la fabrication du sucre" (Paris, 1878).
N. Z.	Neue Zeitschrift für Rübensuckerindustrie.
Oe.	Oesterreichisch-Ungarische Zeitschrift für Zuckerindustrie.
Prager Marktb.	Prager Marktbericht.
S. C.	The Sugar Cane.
S. ind.	La sucrerie indigène et coloniale.
St. J.	Stammer's "Jahresbericht der Zuckerfabrikation."
Weber	Weber's "Zeitblatt für Gewerbetreibende."
Woodcroft	Woodcroft's "Abridgments of Specifications relating to Sugar" (London, 1871).
Z.	Zeitschrift des Vereins der Deutschen Zuckerindustrie.
Z. ang.	Zeitschrift für angewandte Chemie.
Z. B.	Zeitschrift für Zuckerindustrie in Böhmen.
Zerban	Zerban's "Louisiana Bulletin No. 103" (Baton Rouge, 1908).

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